

MSC-A-D-66-3
REVISION A



NATIONAL AERONAUTICS AND SPACE ADMINISTRATION

PROCEDURES AND REQUIREMENTS FOR THE
EVALUATION OF SPACECRAFT NONMETALLIC MATERIALS

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REVISION A

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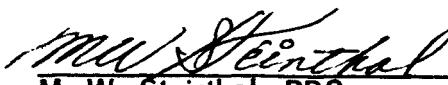
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PROCEDURES AND REQUIREMENTS FOR THE EVALUATION OF SPACECRAFT NONMETALLIC MATERIALS

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SUMMARY

The specifications contained in this document consist of revisions of the standardized test plans of MSC-A-D-66-3 to conform to the new "nonmetallic materials selection guidelines." (Document ASP0-RQTD-D67-5A) which impose more rigid requirements on the selection and performance of Apollo Spacecraft Nonmetallic Materials.

The revised specifications will require an analysis and re-evaluation of the materials currently used. The re-evaluation will consist of reviewing those materials in light of their flammability and offgassing properties, and categorizing them according to their usage as outlined in the D67-5A document. Those which will not meet the criteria of this specification will require that a substitute material be designated as a replacement and tested for conformance to the current requirements. All other materials will be tested if their usage category so dictates, and they will be tested in their usage configuration wherever possible.

The detailed procedures specified in this document may not be replaced by contractor procedures unless they are reviewed by NASA Spacecraft Nonmetallic Materials Selection Review Board and it is ascertained that they will provide the same test conditions for a representative variety of standard materials furnished by MSC.

The test requirements according to the usage category are the following:

1. Upward Combustion Rate
2. Downward Combustion Rate
3. Flash and Fire Points
4. Electrical Wire Insulation and Accessory Flammability
5. Electrical Potting and Coatings Flammability
6. Odor
7. Carbon Monoxide and Offgassing Products
8. Hot Wire Ignition for Vented Containers
9. Electrical Overload Ignition for Hermetically Sealed Containers
10. Simulated Panel & Assembly Flammability (Guidelines)
11. Simulated Crew Bay Configuration Flammability Verification (Guidelines) (MSC responsibility)

Other characteristics may be determined at the option of the contractor, as screening tests or to supplement the required data. Examples are:

- A - Thermogravimetric Analysis with Spark Ignition
- B - Differential Scanning Calorimetry with Spark Ignition
- C - Electrochemical Initiation of Wire Insulation Fire
- D - Autogenous Ignition
- E - Friction and Impact Ignition (Guidelines)
- F - Gap Propagation (Guidelines)
- G - Heat of Combustion

Modifications of the procedures for the supplementary tests must be submitted to NASA for review and concurrence. The test results shall likewise be submitted to NASA for information purposes.

Before testing begins, the contractor must submit to NASA for approval, his list of materials completely identified and accompanied by data from any previous tests. This will preclude duplicate testing by other contractors or agencies.

Also, before experimental results will be accepted, each test facility must demonstrate its ability to reproduce results obtained in other facilities on standard materials specimens. MSC will stock and provide the standards.

All tests will be performed in the environments listed in the table below:

Module	PSIA O ₂ (Flammability)	PSIA O ₂ (Offgassing)
CM	16.5 and 6.2	5.0
LM	6.2	5.0

Exceptions to those testing pressures will be noted in the pertinent category listed in the guidelines (ASPO-RQTD-D67-5A) under the test requirements.

The organic offgassing test criteria now require that the test results show the offgassing rate and the quantity of the offgassed products determined by their contribution to the total contaminant load of the spacecraft exclusive of the quantities removed by the ECS.

This method ensures the optimum limitation of offgassing materials in the spacecraft which is 300 ppm total based on the spacecraft volume at 5.0 psia O₂.

Thus, the use of a material is determined as follows: If a material offgasses the equivalent of 50 ppm a maximum of 6.0 pounds may be used. If other materials already contribute 250ppm, then a maximum of one pound may be used.

Concerning equipment stored in vented containers, in certain cases the materials composing them need not be tested individually for the acceptance requirements, but only the end item. This holds only for equipment stored in vented containers which contain no internal ignition source and for equipment which by its nature must be composed of flammable materials. An example is the food and food packaging.

The reason for igniting the materials in vented containers from the inside is to determine, in the event a material from such a container become ignited, whether stuffing the burning item back into the container would propagate the flame throughout the container with the evolution of flame and heat to adjacent materials and equipment.

Of special note is the Simulated Crew Bay Configuration Flammability Verification Tests to be performed at MSC. This test commences after the materials have passed the standard acceptance tests required by the pertinent usage category.

For these "mockup" tests, an actual expensive piece of equipment need not be used. Electronic and non-flammable parts can be simulated. The "worst case" flammable materials must be configured as closely as possible to the flight item.

"Worst case" means an assembly of many nonmetallic materials, such as Category B types, in close proximity to each other or a larger quantity of one or two materials which might ordinarily fit in Category A. Thus, if a subsystem or system contains glass, ceramic and other nonflammable parts which are potted in foams and resins, then the parts can be simulated but the potting compounds must be configured in the same quantity and place as in the flight unit. This same reasoning holds for hermetically sealed equipment and other similar items.

For Suit Loop materials noted in Category C, no flammability tests are required for those materials used where no ignition source is present in the system. The use of materials in this category must be so justified with pertinent backup data

It is recognized that there are unique applications of materials throughout the spacecraft which applications do not seem to fit a definite category. Inquiries concerning such problems should be directed to the contractor's Nonmetallic Materials Selection Review Board or responsible materials engineer and thence, if necessary, to the prime board at MSC.

Requests for deviations from the context of any requirements contained in the above specifications may be submitted, with complete engineering analysis, to the Nonmetallic Materials Selection Review Board.

NONMETALLIC MATERIALS SELECTION REVIEW BOARD PROCEDURE

I. SCOPE

This procedure will cover all activities by both contractor and NASA-MSC where nonmetallic materials are proposed for use in the spacecraft but do not meet the established selection guidelines per ASPO-RQTD-D67-5A.

II. RESPONSIBILITY

The contractor will bear the primary design responsibility for the selection of the subject materials in CFE as prescribed by appropriate contractual agreements. NASA will bear the responsibility for the review of the selection of certain classes of materials to be herein delineated. Where NASA organizational elements are acting as major contractors, i.e., for GFE, they will submit deviation requests to the MSC Nonmetallic Materials Selection Review Board.

They shall, however, have within their own organization and shall establish at their subcontractors installation one or more cognizant materials engineers who will be responsible for reviewing materials problems, formulating deviation requests and submitting them to the MSC Materials Selection Review Board.

NASA will be responsible for establishing and publishing the general overall materials selection requirements with the contractor being responsible for implementing these requirements. As part of this responsibility, the contractor will be required to provide to NASA for review certain essential elements of information to be delineated here.

The NASA-MSC Board shall have the final responsibility for approval of deviation granted by the contractors' boards including those deviation decisions which have been overridden by the contractors' boards chairmen and program managers.

III. MATERIALS SELECTION REVIEW BOARDS

There will be two types of boards set up. The first or prime board will be the NASA-MSC board located at Houston and chaired by the Chief, RQ&T Division, or his designated alternate, who will have the task of staffing the board with representatives of the technical disciplines concerned with the selection and utilization of spacecraft nonmetallic materials. There should be at least four representatives. The disciplines represented should

include Flight Safety, Space Medicine, Materials Reliability and Quality, and Materials Engineering. The second board will be that one located at the prime contractor's facilities except for NASA installations as noted above. This board shall have as its chairman, the contractor's counterpart to the NASA-MSC board chairman who shall staff his board with the same type members as sit on the NASA-MSC board.

Sub-contractors shall submit their deviation requests to their prime contractor's board.

The prime board shall receive notification of all other boards' activities within 48 hours.

The categories containing materials which may require deviations are the following:

- A. Extensively used materials exposed to crew bay.
- B. Special applications and minor exposed materials in the crew bay.
- C. Materials in suit loop - low pressure 19 psia.
- D. Materials in high pressure O₂ system.
- E. Materials in hermetically sealed containers.
- F. Materials in vented containers.
- G. Materials in nonflight equipment
 - 1) Major exposed
 - 2) Minor exposed
- H. Materials in uninhabited portions of the spacecraft.
 - 1) Major exposed
 - 2) Minor exposed

The basic intent is to have the Contractor's Board assume prime deviation responsibility since it will account for most of the nonmetallic materials. To this end the Contractor Board will either approve or reject all deviation requests and submit a copy of the decision to the NASA Board for review and approval. The submittal will be via datafax from the contractors.

The prime board shall indicate concurrence or non-concurrence to the lower board within five days after receipt of the lower board's activities and decisions. The exception to this will be deviations on Class A materials where no action shall be initiated or approved prior to concurrence by the NASA Board.

For materials selection at subcontractors, the contractor is empowered to establish whatever appropriate review and control mechanisms he deems consistent with the above requirements, and report this action to the NASA Board for its review.

OPERATION OF THE BOARD

A Board meeting will be official when the chairman or his designated alternate and all the delegated members are in session. The Board may be in session either by all being physically present at the same place or linked by other means of communication when physically separated.

The chairman of the board will, in all matters, endeavor to reflect the consensus or will of the majority of the members. He may, override the other board members with his own recommendation if he deems that the circumstances warrant this action. In such cases, the chairman must submit to the Program Manager, with copies to each member of the Board, a written explanation within 24 hours of such an overriding recommendation indicating the circumstances and reasons for his actions. The remaining members of the Board may file with the Program Manager their reasons for differing with the Chairman's decision. The Program Manager shall make the final determination. The Chairman shall disseminate to each Board member a copy of the Program Manager's decision.

Some of the groundrules for override considerations are the following:

1. Effect on -

- a) Crew Safety
- b) Mission Success
- c) Schedule
- d) Funds

2. Alternate procedures.

3. Availability of substitute materials.

Meetings of the board will normally be called and scheduled by the Chairman

who will ensure that an agenda is prepared and that other members are made aware of the agenda at least 24 hours in advance.

The chairman will also be responsible for ensuring that the appropriate records and minutes are kept of the meetings and the decisions rendered by the board and the results of the board meetings are disseminated to all cognizant personnel.

V. SUBMITTAL OF REQUEST FOR DEVIATION

Each board will ensure that an appropriate form is publicized and is used for submitting deviation requests to the board. A form is attached as a suggested sample.

VI. SUPPORTING DATA FOR THE BOARD

Each board will ensure through the appropriate organizations that sufficient supporting data and information are available to guide it in considerable technical depth as to deliberations and decisions. Specifically, it must have available to it a suitable information center which contains data on materials characteristics, on the actual usage of nonmetallic materials in the spacecraft under consideration (such as through use of three-dimensional sketches, etc.), lists of materials indicating their acceptability or unacceptability and behavioral characteristics, motion pictures and photographs as appropriate, and other supporting data.

Return Completed Form to
M. W. Steinthal
PR2 Extension 4546

REQUEST FOR DEVIATION
NONMETALLIC MATERIALS FOR CREW BAY USAGE

DEVIATION NO.
(For Board Use Only)

DATE

Material name, generic (eg. Silicon elastomer nylon fabric, epoxy adhesive, describe chemically if known)

Manufacturers designation (eg. MX 42, RTV 560, EPOW 828)

Manufacturer (eg. Shell Chemical, 3M, Dow Corning. Do not give distributor or user)

Intended use (e.g. Adhesive, insulation coating etc.)

Where to be used (eg. Suit, ECS electrical power, survival, etc. Give name and applicable drawing number)

Amount to be used, weight (Give to $\pm 10\%$.)

Surface area exposed to cabin environment (Give to $\pm 10\%$.)

DEVIATION NUMBER _____

Applicable test data (Enter report number. A copy of all test data required to substantiate approval shall be submitted with the request.)

Applicable specifications (Document used for identification and purchase)

Maximum service temperature

Reason for request, (State reason for material deviation from NASA/MSC requirements eg. odor, toxicity, flammability)

Rationale (Give justification for design adequacy.)

Organization Requesting DEVIATION

Initiators Signature

DEVIATION NUMBER _____

BOARD ACTION

Approved Other

Disapproved

Date

Chairman

Member

Member

Member

Member

NONMETALLIC MATERIALS SELECTION GUIDELINES

I. PURPOSE

The purpose of this document is to establish acceptance guidelines and test requirements for nonmetallic materials in the Apollo spacecraft.

II. SCOPE

The scope of this specification covers:

- A. Major Exposed Materials in the Crew Bay Environment
- B. Special Applications and Minor Exposed Materials in the Crew Bay
- C. Suit Loop Materials
- D. Materials in the High Pressure Oxygen System
- E. Materials in Hermetically Sealed Containers in the Crew Bay Environment
- F. Materials in Vented Containers in the Crew Bay Environment
- G. Materials in Non-Flight Equipment
- H. Materials Outside the Crew Bay Environment

These guidelines shall apply to all spacecraft equipment used during ground tests and all flights. The materials selected for use shall meet the requirements of this document and those documents referenced herein.

III. METHOD OF CATEGORIZATION

Materials shall be categorized primarily by functional application and spatial distribution. This section also defines material toxicity and flammability characteristics as well as test requirements for each category.

A. Category A - Major Exposed Materials in the Crew Bay Atmosphere

This category shall include the material applications that are essentially unlimited with respect to quantity, proximity to ignition sources or exposure to oxygen and are used over wide areas of the spacecraft. To prevent flame propagation, these materials must be self-extinguishing as defined below.

1. Functional Description

Materials performing the following functions shall be subject to the requirements of this category:

- a. Debris traps or nets
- b. Spacesuit outer materials
- c. Couch cushions
- d. Webbing and harness
- e. Spacecraft electrical wire bundles and accessories
- f. Potting compounds and coatings materials

2. Test Requirements

- a. Combustion rate - Self-extinguishing when ignited at the bottom of the test specimen - Test No. 1
- b. Flash & Fire point - Test not required
- c. Odor - Test No. 6
- d. Carbon Monoxide & Offgassing Products Determination - Test No. 7
- e. Electrical Wiring Overload Test - Test No. 4
- f. Connector Potting Electrical Overload Test - Test No. 5

B. Category B - Special Applications and Minor Exposed Materials
in the Crew Bay Atmosphere

This category includes those material applications in the spacecraft arranged in discrete locations and exposed to the cabin atmosphere. The specific amount and arrangement of materials at any discrete locations shall be limited so as to prevent pressure and temperature increases from exceeding allowable limits as a result of combustion. Also, the type of material and/or installation arrangement shall be such that the fire cannot spread to any other nonmetallic material in the pressure vessel. Efforts shall be made to select materials which, upon burning, do not evolve toxic gases.

1. Functional Description

Those materials located and contained as described above and performing the following functions shall be subject to these guidelines:

- a. Electrical connectors including inserts, potting compounds, shell, and mounts.
- b. Electrical components including terminal boards, conformal coatings, and potting compounds.
- c. Instrument panels including control knobs, dial faces, luminescent panels, panel fronts, paints and coatings, and potting compounds.
- d. Batteries and associated equipment.
- e. Miscellaneous materials such as:
 - (1) Discretely isolated shock absorber materials.
 - (2) Discretely isolated thermal insulation materials.
 - (3) Plastic strips.
 - (4) Nonmetallic films and containers.
 - (5) Tapes.
 - (6) Seals and/or sealants.
 - (7) Paint
 - (8) Adhesives.
 - (9) Lubricants
- f. Other material applications - All material applications not included in other categories shall be considered subject to these Category B guidelines and requirements.

2. Test Requirements

- a. Propagation rate - The propagation rate measured downward shall not exceed 0.3 inch per second when tested as outlined in Test No. 2. There shall be no spark, sputter, drip, or transfer of solid mass during burning.
- b. Flash and Fire Point - Test No. 3.
- c. Odor - Test No. 6.
- d. Total Organics and Carbon Monoxide Determination - Test No. 7.
- e. Connector Potting, Electrical Overload Test - Test No. 5.
- f. Panel and Assembly Flammability Tests - Test No. 10.
- g. Gap Propagation - Supplementary Test No. F.

C. Category C - Suit Loop Materials in the Crew Bay Atmosphere

This category shall include those materials used in the suit loop (less than 20 psia oxygen).

1. Functional Description

Materials within the ECS suit loop and interior of space suit such as hoses, helmets, flex joints, couplings, valve seats and seals including regulators and check valves, and inner garment materials, shall be subject to these requirements.

Suit loop wiring and electrical components such as biomedical communications, harnesses, signal conditioners, connectors, transducers, wetness sensors, and thermo-couples, shall also be included

2. Material Property Requirements

a.	Propagation Rate	Self-extinguishing - Test No. 1
b.	Flash and Fire Points	No test required
c.	Total Organic and Carbon Monoxide Determination	Test No. 7
d.	Odor	Test No. 6
e.	Electrical Wiring Overload Test	Test No. 4
f.	Connector Potting, Electrical Overload Test	Test No. 5
g.	Panel and Assembly Flammability Tests	Test No. 10

D. Category D - Materials in High Pressure Oxygen System

This category shall include those materials used in greater than 20 psia oxygen systems. Materials should have prior use or test history in oxygen service with no fire or explosion experience.

1. Functional Description

Materials for such applications as filters, seals, valve seats, and pressure bladders, shall be covered by this criteria.

2. Test Requirements

a.	Propagation Rate	No test required
b.	Flash and Fire Points	Test No. 3
c.	Odor	Test No. 6
d.	Total Organic and Carbon Monoxide Determination	Test No. 7
e.	Friction and Impact Ignition	Supplementary Test No. E.

E. Category E - Material Applications in Hermetically Sealed Containers

This category shall include those material applications inside hermetically sealed containers (with inert gas, air, and/or potting with no method of receiving or replenishing a supply of oxygen).

1. Functional Description

Applications such as electrical wiring, tie wraps, potting compounds, and electronic components shall be subject to these guidelines.

2. Test Requirements

The sealed container shall not rupture as a result of internal temperature or pressure increases caused by internal electrical malfunction. Verification by test that a worst case configuration meets this criteria shall be required. Rationale shall be furnished to support the selected test configuration.

- a. Test selection criteria** - Tests of representative worst case configurations of hermetically sealed containers containing ignition or heat sources shall be conducted to verify acceptability. The contractor shall provide analysis to support the selection of test configuration.
- b. Test Configuration** - The equipment tested shall simulate the flight article except for the "worst case" flammable materials which shall be identical to the flight item in quantity and placement.
- c. Test Conditions and Procedures** - Test No. 9.
- d. Acceptance Criteria** - The hermetically sealed container shall not rupture or exceed design leakage requirements when tested according to the above requirements.

F. Category F - Materials in Vented Containers

This category shall include those materials used inside unsealed containers with or without an internal ignition source. The containers shall allow over-pressure to be vented and a fire inside shall not propagate outside the container. Removal of materials from containers shall be permitted for operational use only.

1. Functional Description

Applications where an internal ignition source is present shall include foams, printed circuit boards, wiring, potting compounds, and electronic components.

Applications embodying no internal ignition source shall include food bags, survival gear, and personal equipment.

2. Test Requirements

a.	Combustion Rate	No test required
b.	Flash and Fire Point	Test No. 3
c.	Odor	Test No. 6
d.	Total Organic and Carbon Monoxide Determination	Test No. 7
e.	Hot Wire Test for Vented Containers With No Internal Ignition Source	Test No. 8
f.	Electrical Wire Insulation and Accessory Flammability Test for Vented Containers With an Internal Ignition Source	Test No. 4
g.	Panel and Assembly Flammability Tests	Test No. 10

G. Category G - Materials Applications in Non-Flight Equipment

This category shall include those materials used in non-flight equipment during closed hatch, power-on tests.

Materials used in manned oxygen atmosphere tests must meet applicable Category A through F tests and requirements.

1. Functional Description

Applications shall include "Remove Before Flight" flags, plastic bags, plastic tape, carry-on GSE, and associated cables.

2. Test Requirements for Air Exposure Only

a. Propagation Rate

- 1) Major Exposed Materials Self-Extinguishing Test No. 1
- 2) Minor Exposed Materials Test No. 2

b. Flash and Fire Points

- 1) Major Exposed Materials No test required
- 2) Minor Exposed Materials Test No. 3

c. Odor Test No. 6

d. Carbon Monoxide and Offgassing Products Test No. 7

H. Category H - Materials in Uninhabited Portions of the Spacecraft

The material applications included in this category shall be those in uninhabited portions of the spacecraft simultaneously exposed to ignition sources and an atmosphere other than pure oxygen.

1. Functional Description

Applications shall include insulation blankets, protective covers, wire bundles and harness, tape, conformal coating, and encapsulants.

2. Test Requirements

a. Propagation Rate

1) Major Exposed Materials Self-Extinguishing in air
Test No. 1

2) Minor Exposed Materials Test No. 2 (in air)

b. Flash and Fire Points

1) Major Exposed Materials No test required

2) Minor Exposed Materials Test No. 3 (in air)

c. Odor No test required

d. Carbon Monoxide and Offgassing Products No test required

e. Electrical Wiring Overload Test No. 4 (in air)

f. Connector Potting Electrical Overload Test No. 5 (in air)

TEST NO. 1

PROPAGATION RATE TEST

UPWARD

1.0 PURPOSE

This procedure is designed to screen spacecraft crewbay materials to detect and remove those which could promote the rapid spread of fire.

2.0 TEST CONDITIONS - PRESSURES

The pressure environments for the pertinent category materials shall be selected from the following table:

<u>Category</u>	<u>Module</u>	<u>PSIA</u>
A	CM	16.5 and 6.2 Oxygen
A	LM	6.2 Oxygen
C	CM and LM	19.0 Oxygen
G	CM and LM	16.5 Oxygen
H	CM and LM	14.7 Performance Atmosphere

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have a QA coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test conduct shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

Materials shall be self-extinguishing when ignited at the bottom of the test specimen.

5.0 TEST EQUIPMENT

5.1 Test chamber shall have a volume sufficient to provide 12 liters per gram of sample materials and suitably constructed and protected to insure safe operation. A window or viewing port for visual observations shall be included. The test chamber shall contain inlets for vacuum, an ignition wire, air, and oxygen. The chamber is to be fully protected against the possibility of operator injury in the event of explosive rupture.

Organic materials used in the construction of the chamber such as gaskets and seals shall be of types which contribute little or no outgassing to the chamber or which can be pre-outgassed by vacuum cycling to a minimal identifiable amount, i.e., less than 10Oppm based on the chamber volume. A vertical sample holder shall be included and positioned within the test chamber.

5.2 Pressure Gauge - A pressure gauge capable of measuring pressures from 0.1 to 30 psia with an accuracy of ± 0.1 psia, or a pressure transducer and recorder with comparable capability shall be used.

5.3 Oxygen Supply - The oxygen shall be commercially available oxygen conforming to specification MIL O-26210, Type 1. Efficient and safe equipment shall be used for measuring the flow and for transferring the oxygen to the test chamber.

5.4 Sample Holder - The sample holder shall consist of a vertically mounted steel clamp which overlaps one fourth inch in each side of a specimen along the full five inch length of the sample, leaving a two inch wide by five inch long exposed center section.

5.5 Ignition Source - Ignition of the sample shall be accomplished by employing a regulated energy flux. The ignition source shall be a nichrome wire wrapped around its center portion with a 1" x 2" piece of facial type tissue and placed in contact with the bottom edge of the sample. The power supply to the wire shall provide sufficient voltage, controlled by means of a variable transformer, to ignite the tissue.

5.6 Propagation Rate Indicators

5.6.1 Motion Pictures - Motion picture records shall be kept of each burning test.

6.0 SAMPLE PREPARATION

- 6.1 All material specimens shall be free of cuts, abrasions, or other flaws as determined by close visual inspection. Before the test the samples shall be cleaned by brushing or by flowing an inert gas over them to remove loose surface contamination.
- 6.2 Films and fabrics shall be tested in their "as received" condition. Specimens shall be cut out in the form of rectangles two and one half inches wide and five inches long. Foams or other thick materials shall be used in the applied thickness and be two and one half inches wide and five inches long.
- 6.3 Primers, coating materials, and paints shall be applied on the material actually used in the spacecraft whenever possible. The coatings shall be applied in a thickness equivalent to normal practice of use and post cured in accordance with prescribed manufacturing practices.
- 6.4 Materials and components which will be used in an irregular size or shape shall be tested in the "as purchased" configuration. They shall be attached to the sample holder by fiberglass or metal threads.

7.0 PRETEST PROCEDURE

- 7.1 Verify that all test equipment is in current calibration.
- 7.2 Witness motion picture camera setup.
- 7.3 Verify oxygen certification (MIL 0-27210, Type 1).
- 7.4 Verify material identification as one of the following:
 - 7.4.1 Manufacturer's Certification
 - 7.4.2 NASA Certification
 - 7.4.3 Contractor Certification
 - 7.4.4 Definite identification not available.

- 7.5 Prepare three samples per appropriate paragraph in section 5 above.
- 7.6 If irregularly shaped samples are tested describe the shapes.
- 7.7 Visually inspect each sample. (There shall be no cuts, abrasions or other flaws.)
- 7.8 Clean samples by brushing or by flowing an inert gas to remove loose surface contamination.
- 7.9 Weigh the samples and record the weight.
- 7.10 Record the volume of the test chamber in liters.
- 7.11 Verify the test chamber has a volume equal to or greater than 12 liters per gram of sample material.
- 7.12 Mount the sample in the sample holder and verify that the exposed center section is 2.0 ± 0.1 inches wide.
- 7.13 Position sample holder within the chamber.
- 7.14 Place the ignition wire horizontally in contact with or not more than one eighth inch away from the sample at the midpoint of its two inch width at the bottom.

8.0 TEST PROCEDURE

- 8.1 Evacuate the chamber to less than five (5) Torr and continue pumping the chamber for five (5) minutes .
- 8.2 Isolate the chamber and monitor pressure for one (1) minute. Testing may not begin until all leaks are corrected. (A leak is indicated if an increase in pressure of more than one (1) Torr occurs.
- 8.3 Pressurize the chamber to the required psia with oxygen.
- 8.4 Analyze the chamber environment to verify that a minimum of 95.0 percent oxygen exists. Repurge or bake out chamber until this condition is met. (Record last oxygen percent reading.)

- 8.5 After the chamber has stabilized at the test psia, soak the samples as follows: Ten (10) minutes for dense materials and thirty (30) minutes for porous materials. Record soak time.
- 8.6 Verify chamber pressure is the test psia and isolate the chamber.
- 8.7 Start Motion Picture Camera and other applicable instruments.
- 8.8 Apply current to ignition wire.
- 8.9 Record whether sample is self extinguishing.
- 8.10 Note combustion characteristics (nature and color of flame, soot, residue and other pertinent observations).
- 8.11 Determine by analytical means, such as those employed in Test No. 7, the actual gaseous products of combustion.
- 8.12 Record the rate of pressure rise and the final pressure in the chamber.
- 8.13 Secure the chamber.

9.0 REPORTING

- 9.1 Name of the material (generic).
- 9.2 Vendor designation and vendor.
- 9.3 Self extinguishing (yes or no).
- 9.4 Combustion Characteristics - Distance that flame progressed before extinguishing, flame phenomena and temperature, etc.
- 9.5 Identification and qualification of combustion products.
- 9.6 Rate of pressure rise and final pressure.
- 9.7 Disposition or status, dimension, and size of sample material.
- 9.8 Name and number of test procedure.

9.9 Date of test.

9.10 Test Number.

9.11 Identify of the testing agency or component.

9.12 Name of test conductor.

TEST NO. 2
DOWNWARD PROPAGATION RATE TEST

1.0 PURPOSE

This procedure is designed to screen spacecraft crew bay materials to detect and remove those which could promote the rapid spread of a beginning fire.

2.0 TEST CONDITIONS - PRESSURES

This test shall be performed for all Category "B" materials. If any material tested in 16.5 psia oxygen fails to meet the combustion requirements, it will be retested at 6.2 psia and the results reported.

All Category "B" materials used in the Lunar Module shall be tested at 6.2 psia oxygen and the results reported.

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test conduct shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

The propagation rate measured downward shall not exceed 0.3 inches/sec. There shall be no spark, sputter, drip or transfer of solid mass during burning.

5.0 TEST EQUIPMENT

- 5.1 Test chamber shall have a volume sufficient to provide 12 liters per gram of sample materials and suitable constructed and protected to insure safe operation. A window or viewing port for visual observation shall be included. The test chamber shall contain inlets for vacuum, an ignition wire, air, and oxygen. The chamber is to be fully protected against the possibility of operator injury in the event of explosive rupture.

Organic materials used in the construction of the chamber such as

gaskets and seals shall be types which contribute little or no outgassing products to the chamber or which can be pre-outgassed by vacuum cycling to a minimal identifiable amount, i.e., less than 10ppm based on the chamber volume. A vertical sample holder shall be included and positioned within the test chamber.

- 5.2 Pressure Gauge - A pressure gauge capable of measuring absolute pressures from 0.1 to 30 psia with an accuracy of ± 0.1 psia, or a pressure transducer and recorder with comparable capability shall be used.
- 5.3 Oxygen Supply - The oxygen shall be commercially available oxygen conforming to specification MIL 0-27210, Type 1. Efficient and safe equipment shall be used for measuring the flow and for transferring the oxygen to the test chamber.
- 5.4 Sample Holder - The sample holder shall consist of a vertically mounted steel clamp which will overlap one fourth inch in each side of a specimen along the full five inch length of the sample, leaving a two inch wide by five inch long exposed center section.
- 5.5 Ignition Source - Ignition of the samples shall be accomplished by employing a regulated energy flux. The ignition source shall be a nichrome wire wrapped around its center portion with a 1" x 2" piece of facial type tissue and placed in contact with the bottom edge of the sample. The power supply to the wire shall provide sufficient voltage, controlled by means of a variable transformer, to ignite the specimen.
- 5.6 Propagation Rate Indicators
 - 5.6.1 High Speed Movie - A high speed movie shall be kept of each burning test. The equipment shall be calibrated to assure that burning rates can accurately be determined from the films.
 - 5.6.2 In addition to motion pictures, supporting data from a vertical bank of thermocouple indicators combined with a recorder may be used. However, a precision of at least five percent shall be obtained with the measuring device. If thermocouples are used a minimum of four thermocouples shall be installed. Loss of more than

one thermocouple or loss of either end point thermocouple will invalidate the test.

6.0 SAMPLE PREPARATION

- 6.1 All material specimens shall be free of cuts, abrasions, or other flaws as determined by close visual inspection. Before the test the samples shall be cleaned by brushing or by flowing an inert gas to remove loose surface contamination.
- 6.2 Films and fabrics shall be tested in their "as received" condition. Specimens shall be cut out in the form of rectangles two one half inches wide and five inches long. Foams or other thick materials shall be tested in the applied thickness and be two and one half inches wide and five inches long.
- 6.3 Primers, coating materials, and paints shall be applied on the material actually used in the spacecraft whenever possible. The coatings shall be applied in a thickness equivalent to normal practice of use and post cured in accordance with prescribed manufacturing practices.
- 6.4 Materials and components which will be used in an irregular size or shape shall be tested in the "as purchased" configuration. They shall be attached to the sample holder by fiberglass or metal threads.

7.0 PRETEST PROCEDURE

- 7.1 Verify that all test equipment is in current calibration.
- 7.2 Witness high speed movie camera setup.
- 7.3 Witness thermocouple setup and checkout.
- 7.4 Verify oxygen certification (MIL 0-27210, Type 1).
- 7.5 Verify material identification as one of the following:
 - 7.5.1 Manufacturer's Certification
 - 7.5.2 NASA Certification
 - 7.5.3 Contractor Certification

7.5.4 Definite identification not available.

7.6 Prepare three samples per appropriate paragraph of Section 5 above.

7.7 If irregularly shaped samples are tested describe the shapes.

7.8 Visually inspect each sample. (There shall be no cuts, abrasions or other flaws.)

7.9 Clean samples by brushing or by flowing an inert gas to remove loose surface contamination.

7.10 Weigh the samples and record the weight.

7.11 Record the volume of the test chamber in liters.

7.12 Verify that the test chamber has a volume equal to or greater than twelve liters per gram of sample material.

7.13 Mount the sample in sample holder and verify that the exposed center section is 2.0 ± 0.1 inches wide.

7.14 Position sample holder within the chamber.

7.15 Place the ignition wire horizontally in contact with, or not more than one eighth inch away from the sample at the midpoint of its two inch width at the top.

8.0 TEST PROCEDURE

8.1 Evacuate the chamber to less than five (5) Torr and continue pumping the chamber for five (5) minutes.

8.2 Isolate the chamber and monitor pressure for one (1) minute. Testing may not begin until all leaks are corrected. (A leak is indicated if an increase in pressure occurs.)

8.3 Pressurize the chamber to 16.5 ± 0.1 (or 6.2 ± 0.1) psia with oxygen.

8.4 Analyze the chamber environment to verify that a minimum of 95.0 percent oxygen exists. Repurge or bake out chamber until this condition is met. (Record last oxygen percent reading.)

- 8.5 After the chamber has stabilized at 16.5 ± 0.1 (or 6.2 ± 0.1) psia, soak the samples as follows: Ten (10) minutes for dense materials and thirty (30) minutes for porous materials. Record soak time.
- 8.6 Verify chamber pressure is 16.5 ± 0.1 (or 6.2 ± 0.1) psia and isolate the chamber.
- 8.7 Start propagation rate measurement and other applicable instruments.

*See Paragraph 3.0 for pressure changes.

NOTE: Stop watch is to be started at the first visual indication of combustion and stopped when the flame front reaches the sample base.

- 8.8 Apply current to ignition wire.
- 8.9 Record peak chamber pressure after ignition.
- 8.10 Record the combustion rate determined by Visual Method using a stopwatch.

<u>Sample No. 1</u>	<u>Sample No. 2</u>	<u>Sample No. 3</u>	<u>Sample Length</u>
Combustion Time (Sec) <u>From Ignition</u>	Combustion Time (Sec) <u>From Ignition</u>	Combustion Time (Sec) <u>From Ignition</u>	
_____	_____	_____	5 Inches

- 8.11 Note combustion characteristics (nature and color of flame, soot, residue and other pertinent observations).

9.0 POST TEST PROCEDURES

- 9.1 Record propagation rate determined by the Photographic Method.
- 9.2 Record propagation rate determined by the Thermocouple Method.
- 9.3 Comments and Observations.

10.0 REPORTING

Unless otherwise specified the following test data and pertinent information concerning the materials shall be reported.

- 10.1 Name of material (generic).
- 10.2 Vendor designation and vendor.
- 10.3 Propagation rate in inches per second.
- 10.4 Combustion characteristics.
- 10.5 Rate of pressure rise and final pressure.
- 10.6 Identification and quantification of combustion products.
- 10.7 Disposition or status, dimension, and size of sample material.
- 10.8 Name and number of test procedure.
- 10.9 Date of test.
- 10.10 Test number.
- 10.11 Identity of the testing agency or component.
- 10.12 Name of test coordinator.

TEST NO. 3
FLASH POINT AND FIRE POINT TESTS

1.0 PURPOSE

This test is designed to evaluate the spark ignition characteristics of nonmetallic materials which are to be used in the areas of manned spacecraft exposed to the pure oxygen environments.

2.0 TEST CONDITIONS - PRESSURES

This test shall be performed for all category B, D and F materials. If any material fails to meet the requirements when tested at 16.5 psia oxygen, it shall be retested at 6.2 psia and the results reported.

<u>Categories</u>	<u>Module</u>	<u>PSIA (oxygen)</u>
B, D, F	CM	16.5 & 6.2
B, D, F	LM	6.2

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

4.0 DEFINITIONS

- 4.1 Flash Point - The flash point is the lowest temperature at which a material will give off, at or near its surface, flammable vapor which, when mixed with oxygen or air in the environment and exposed to an ignition source, will provide a nonself-sustaining flash or flame.
- 4.2 Fire Point - The fire point is the lowest temperature at which the mixture of vapors from the surface of a material and air or oxygen continue to burn after ignition. A self-sustaining, self-propagating glow shall be considered equivalent to flaming combustion.

5.0 CRITERIA OF ACCEPTABILITY

- 5.1** A material that evolves vapors which produce a visible flash at a temperature of less than 400°F when tested accordingly to Section 8.0 of this specification shall be considered unacceptable for use in the manned spacecraft crew bay areas. One or more of the three test specimens failing to meet the above requirements shall constitute the rejection of the materials being tested.
- 5.2** A material that shows evidence of charring, self-sustaining combustion, or other signs of pyrolysis at a temperature less than 450° shall be considered unacceptable for use in the manned spacecraft crew bay areas.

6.0 TEST EQUIPMENT

6.1 Test Chamber

6.1.1 Construction

6.1.1.1 The test chamber shall be suitably constructed to ensure safe operation. A window or viewing port for visual observations shall be included. The chamber is to be fully protected against the possibility of operator injury in the event of explosive rupture.

6.1.1.2 Organic materials used in the construction of the chamber such as gaskets and seals shall be of types which contribute little or no outgassing products to the chamber or which can be pre-outgassed by vacuum cycling to a minimal amount of identifiable vapor, i.e., less than 10ppm based on the chamber volume.

6.1.1.3 The chamber volume shall be large enough to accomodate a sample of a size specified in the appropriate paragraph of Section 7.

6.1.1.4 The test chamber may be heated by any externally applied method desired. However, the heating method must permit a temperature rise of at least 25°F per minute at 200°F and

must maintain a temperature over the test chamber uniform within 10°F after a one minute hold at temperature of 600°F.

- 6.1.2 Thermocouple - All temperature measurements made in the interior atmosphere of the test chamber shall be made using suitable thermocouples. Temperatures shall be measured at one point on the test face of the specimen approximately one half inch from the sparking device.
- 6.1.3 Spark Ignition Equipment
 - 6.1.3.1 Spark needles shall be made of platinum, platinum-rhodium alloy or platinum-iridium alloy wire shaped to a 0.004 inch radius point. The spark gap shall be adjusted to an electrode gap of one eighth inch at the beginning of any test.
 - 6.1.3.2 Spark energy to be supplied by a high voltage capacitor. The capacitor shall be charged with a high voltage DC power supply to provide a spark energy of 40-100 millijoules to break down the dielectric gap between the electrodes. The sparking circuit shall be of minimum convenient capacitance, inductance, and resistance.
- 6.1.4 Oxygen Systems - The oxygen delivery system of the chamber shall consist of corrosion resistant steel tubing of suitable size inside the test chamber. The oxygen shall be brought into the chamber through this tube and exhausted directly from the chamber by the same type or any safe and efficient equipment.
- 6.1.5 Temperature Monitoring - Thermocouple readings may be monitored by multiple point recorders or any equally accurate method. Desired accuracy is $\pm 10^{\circ}\text{F}$.
- 6.1.6 The flash and fire points shall be observed visually in a darkened room or with the aid of a photo cell.

7.0 TEST SAMPLE PREPARATION

- 7.1 Bulk materials shall be in the form of rectangular sheet stock 1 x 4 x 0.030 inches, unless otherwise specified.
- 7.2 Coatings and finishes shall be applied, in the form and thickness as used in the spacecraft, on one side of a 1 x 6 x 0.020 inch rectangular coupon of unclad aluminum. Pretreatment of aluminum and coating application technique shall conform to established practice for the coating test.
- 7.3 Film and fabric material shall be tested in their "as purchased" thickness with length and width dimensions of four inches and one inch, respectively.
- 7.4 Foams shall be in the form of rectangles 1 x 6 x 0.5 inches.
- 7.5 Electrical wire insulation specimens shall be in the form of insulated wire, six inches long. The insulation shall extend the full length of the wire.
- 7.6 Materials not suited to testing in the forms described above shall be tested in special form. In any event, the test specimen weight shall not exceed 5 grams per liter of chamber volume and the specimen configuration shall be recorded.
- 7.7 The specimens shall be cleaned by wiping with a solvent inert to the surface of the test material and drying by blowing with dry nitrogen or oxygen. No evidence of residue shall remain.

8.0 TEST PROCEDURE

- 8.1 The test specimens shall be conditioned by exposure to an atmosphere of 95 percent purity oxygen at (16.5 psia) 855±5 Torr for at least ten minutes for dense materials and thirty minutes for porous materials at room temperature.
- 8.2 The test chamber shall be thoroughly cleaned before each test. "Black Light" may be used to detect the presence of residue.
- 8.3 The specimen shall be mounted in the test chamber with its longest dimension vertical. The sparking point of the ignition needles shall be placed .031 ± .005 inches from the surface of the top of the specimen.

- 8.4 The test chamber shall be closed and evacuated, then filled with oxygen, 95 percent purity conforming to MIL 0-27210, Type 1. Evacuating and filling shall be continued until a calculated 99.4 percent or higher oxygen concentration of the purity noted above exists in the system.
- 8.5 The chamber shall then be checked for leaks by checking the pressure gauge for an increase in pressure. A rise of 1 Torr after a one minute hold denotes a leak which must be corrected before proceeding to the next step.
- 8.6 At the conclusion of the leak test, chamber heating power, sufficient to cause the internal gas temperature to rise at the rate of $25^{\circ}\pm2^{\circ}\text{F}$ per minute, shall be applied. The temperature shall be raised continuously at this rate with sparking at the end of each 25° rise.
- 8.7 The one minute sparking intervals shall be repeated and continued until the flash point has been observed or a maximum temperature of 600°F has been attained.
- 8.8 The test will be terminated when any indication of ignition of vapors of sample is observed.
- 8.9 The fire point tests shall be a continuation of the flash point test. Where flash does not precede fire, the flash point determination will not be considered void.
- 8.10 Testing of fire and flash points shall be stopped when the maximum temperature of 600°F has been reached.
- 8.11 If the behavior of the enclosed volume around the spark indicates by repeated strong flashes of igniting gases, that dangerous quantities of potentially explosive gases are being evolved, the test shall be discontinued.
- 8.12 Three samples of each material shall be tested.

9.0 REPORT

- 9.1 The temperature at which vapors ignite with a non-self-sustaining flame and no evidence of pyrolysis of the surface of the material shall be recorded as the flash point.
- 9.2 The temperature at which self-sustaining, self-propagating flame or glow is apparent on the surface of the material shall be recorded as the fire point.
- 9.3 The report shall include the following information:
 - 9.3.1 Name of the material (generic).
 - 9.3.2 Vendor designation and vendor.
 - 9.3.3 Usage, quantity, and surface area in the spacecraft.
 - 9.3.4 Flash and fire points, combustion characteristics.
 - 9.3.5 Disposition or status, dimensions, and size of sample material.
 - 9.3.6 Name and number of test procedure.
 - 9.3.7 Date of test.
 - 9.3.8 Test number.
 - 9.3.9 Identity of the testing agency or component, and name of test coordinator.

TEST NO.4
ELECTRICAL WIRE INSULATION AND ACCESSORY
FLAMMABILITY TEST

1.0 PURPOSE

This test is designed to screen flammable wire insulation and electrical accessory materials which may be used in the habitable areas of manned spacecraft. Electrical accessory materials consist of wire bundle sleeving, heat shrinkable tubing, bundle ties, cable clamps, identification tags, and coating materials.

2.0 TEST CONDITIONS - PRESSURES

The materials shall be tested at the pressures indicated in the table below for the pertinent categories indicated:

<u>CATEGORY</u>	<u>MODULE</u>	<u>PSIA</u>
A	CM	16.5 + 6.2 (Oxygen)
A	LM	6.2 (Oxygen)
C	CM	19.0 (Oxygen)
C	LM	19.0 (Oxygen)
H	CM	14.7 (Performance Atmosphere)

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

Electrical wire insulation and accessories shall not be capable of sustaining combustion in the designated oxygen pressure test atmosphere following removal of the ignition source with current overloads up to the melting point of the electrical wire.

5.0 TEST EQUIPMENT

- 5.1 Test Chamber - Test Chamber shall have sufficient volume to insure complete combustion of the wire bundle specimen and shall be suitably constructed and protected to insure safe operation. A window or viewing port for visual observations shall be provided. The test chamber shall contain inlets for vacuum, air, and oxygen, and a source of power for wire overload. A horizontal sample holder shall be included and positioned within the test chamber.
- 5.2 Pressure Gauge - The pressure gauge shall be capable of measuring pressure up to 30 psia (1550 Torr) with an accuracy of ± 0.1 psia. A comparable pressure transducer may be used.
- 5.3 Oxygen Supply - The oxygen shall be commercially available oxygen conforming to specifications MIL-0-27210, Type 1. Suitable equipment for transferring oxygen to the test chamber shall be provided. A calibrated method of sampling the chamber atmosphere to verify percent oxygen is required.
- 5.4 Sample Holder - The sample holder shall consist of two horizontally mounted electrical connections, i.e., knurled bolts, spaced twelve inches apart. A non-flammable center support shall be provided to support the center of the sample wire bundle. The electrical terminals shall be connected to the ignition power source.
- 5.5 Ignition Source - An external electrical power supply shall be provided which is capable of providing a large steady current through one wire of the sample bundle so that a very high temperature will be achieved quickly. The power source must be capable of supplying a current 10 percent above the nominal fusion current for that gauge wire being tested.

<u>Wire Gauge No.</u>	<u>Nominal Fusion Current (Amps)</u>
28	15
26	20
24	30
22	40
20	60
18	80
16	120
14	160
12	225

6.0 SAMPLE PREPARATION

6.1 Wire Insulation Samples

- 6.1.1 Insulated wire samples shall be free of cuts, abrasions, or other flaws as determined by close visual inspection.

6.1.2 A test bundle of seven insulated wires, six of which are twelve inches in length and the seventh thirteen inches in length, shall be bound together using portions of the type of wire to be tested in three places four inches apart. The thirteen inch length of wire shall be positioned on the exterior of the bundle and shall be stripped of one half inch of insulation on each end.

6.2 Electrical Wire Accessories

6.2.1 Accessory specimens shall be free of cuts, abrasions or other flaws as determined by close visual inspection.

6.2.2 Accessory specimens shall be installed over or adjacent to wire bundles, prepared per Paragraph 5.1.2, in the manner in which they are intended to be installed in the spacecraft.

7.0 PRETEST PROCEDURE

7.1 Verify that all test equipment is in current calibration.

7.2 Verify oxygen certification (MIL-0-27210, Type 1).

7.3 Verify material identification as one of the items below:

- 7.3.1 Manufacturers Certification.
- 7.3.2 NASA Certification.
- 7.3.3 Contractor Certification.
- 7.3.4 Definite identification not available.

7.4 Visually inspect each sample. (There shall be no cuts, abrasions or other flaws.)

7.5 Prepare three samples per 6.1.2.

7.6 Prepare wire accessories per 6.2.2.

7.7 Record wire gauge number.

7.8 Record power supply current capability in amps. Power supply must meet the requirement of 5.5 for the gauge wire being tested.

7.9 Record thickness of wire insulation in mils.

7.10 Record volume of chamber in liters.

7.11 The sample bundle shall be mounted in the horizontal sample holder and positioned within the test chamber by fastening one end of the stripped wire to each of the electrical connection posts. It shall be supported in the middle by a center support, constructed with the same wire used for the test.

8.0 TEST PROCEDURE

- 8.1 Evacuate the chamber to less than five (5) Torr and continue pumping the chamber for five (5) minutes.
- 8.2 Repressurize the chamber to 16.5 ± 0.1 psia (854 Torr) with oxygen. Allow the chamber to stand for one minute. A leak is indicated if a decrease in test chamber of greater than 0.1 psia is observed after the system is closed off. If leak occurs, the chamber will be brought to atmospheric pressure, leak corrected, and steps 8.1 and 8.2 repeated.
- 8.3 Analyze the chamber environment to verify that a minimum of 95 percent oxygen exists. Repurge or bake out chamber until this condition is met. (Record last oxygen percent reading.)
- 8.4 After the chamber has stabilized at 16.5 ± 0.1 psia soak the specimen for 30 minutes. Record soak time.
- 8.5 Verify chamber pressure is 16.5 ± 0.1 psia and isolate the chamber.
- 8.6 Apply a current of 40 amps to the wire. If ignition is not obtained in one minute, the current shall be increased in 5 amps steps at one minute intervals until the wire fails or ignition occurs. Record whether ignition occurs or wire failed.
- 8.7 Record current level at which ignition occurred or the wire failed.

9.0 REPORTING

Unless otherwise specified the following test data and pertinent information concerning the materials shall be reported.

- 9.1 Name of material.
- 9.2 Vendor designation and vendor.
- 9.3 Results of test.
 - 9.3.1 Combustion characteristics and phenomena
 - 9.3.2 Pressure rise in the chamber.
 - 9.3.3 Current level at end of test.

- 9.4 Name and number of test procedure.
- 9.5 Date of test.
- 9.6 Test number.
- 9.7 Identity of the testing agency or component.
- 9.8 Name of test coordinator.

TEST NO. 5
ELECTRICAL POTTING AND COATINGS

FLAMMABILITY TEST

1.0 PURPOSE

This test evaluates the flammability characteristics of spacecraft potting compounds in a specific oxygen environment. It is designed to simulate a short circuit or dielectric breakdown of current carrying wires, or connector contacts within the potting used to environmentally seal electrical connectors.

2.0 TEST CONDITIONS - PRESSURES

The test chamber pressures shall be selected from the table below as indicated for the category of the material being tested.

<u>Category</u>	<u>Module</u>	<u>PSIA</u>
A	CM	16.5 & 6.2 (oxygen)
A	LM	6.2 (oxygen)
B	CM	16.5 & 6.2 (oxygen)
B	LM	6.2 (oxygen)
C	CM	19.0 (oxygen)
C	LM	19.0 (oxygen)
H	CSM-LM	performance atmosphere

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

Connector potting material shall not be capable of sustaining combustion in the designated oxygen pressure test atmosphere following removal of the ignition source with current overloads up to the melting point of the conductor of the electrical wire.

5.0 TEST EQUIPMENT

- 5.1** Test Chamber - Test chamber shall have sufficient volume to ensure complete combustion of the wire bundle specimen and shall be suitably constructed and protected to ensure safe operation. A window or viewing port for visual observations shall be provided. The test chamber shall contain inlets for vacuum, source of power for wire overload, air, and oxygen. A horizontal sample holder and a central connector mount shall be included and positioned within the test chamber. See Figure 1.
- 5.2** Pressure Gauge - The pressure gauge shall be capable of measuring pressure from 1 to 30 psia (1550 Torr) with an accuracy of $\pm .1$ psia.
- 5.3** Oxygen Supply - The oxygen shall be commercially available with an analytical content of 95 percent or greater conforming to specification MIL 0-27210, Type 1.
- 5.4** Sample Holder and Connector Mount - The sample holder shall consist of two horizontally mounted electrical connections (bolt with knurled nuts) spaced 12 inches apart. The electrical terminals shall be connected to the ignition power source. A central connector mount consisting of a vertical panel drilled to receive a horizontal Bendix PT07 Jam Nut Receptacle shall be provided.
- 5.5** Ignition Source - An external power supply shall be provided which is capable of providing a large steady DC current so that a very high temperature will be achieved quickly. The power source must be capable of supplying 100 amperes of current through a 18 AWG fourteen inch long wire. A photocell to interrupt current at ignition shall be provided.

6.0 SAMPLE PREPARATION

- 6.1** Prepare three (3) samples for each candidate potting compound per paragraphs 6.2 through 6.7 below.
- 6.2** A fourteen inch (14") length of AWG 18 white Teflon insulated wire (MIL-W-16878, Type E, or MIL-W-22759, Type MS21985) shall be prepared as follows:
 - 6.2.1** Form a "U" bend in the middle of the wire and etch the center 4 inch with "Tetra-etch" (W. L. Gore and

Associates, Newark, Delaware). Neutralize in naptha solvent or ethyl alcohol.

- 6.2.2 Strip one half inch insulation from each end.
- 6.2.3 Cut the wire in half such that two inches of etched insulation remains on the unstripped end of each seven inch length.
- 6.3 Obtain a Bendix Pygmy PT06CE-18-11P Straight Plug. Strip 1/4" from the end of each 7" wire prepared per 6.2. Crimp one 7" wire to contact K and the second 7" wire to adjacent contact L.
- 6.4 Crimp nine (9) etched white AWG 20 Teflon insulated wires, each 3" long, in the remaining contacts.
- 6.5 Place the potting boot on the connector and fill with the candidate potting compound, per the manufacturer's instructions. Ensure that the potting compound is within recommended shelf life. Ensure that all steps are followed exactly as they would be on flight hardware, including cleaning and priming of connector rear insert for bondability, degassing potting compound, proper humidity control, etc.
- 6.6 Cure per applicable user's procedure or manufacturer's recommended time/temperature for optimum properties.
- 6.7 Remove potting boot.

7.0 CENTRAL CONNECTOR PREPARATION

- 7.1 Prepare three (3) Central Connectors per paragraphs 7.2 through 7.5 below.
- 7.2 Obtain a Bendix Pygmy PT07CE-18-11S Jam Nut Receptacle. Crimp a short AWG 16 Teflon insulated jumper between contact K and contact L.
- 7.3 Place the potting boot on the connector and fill with the compound to be tested.
- 7.4 Cure per manufacturer's instruction.
- 7.5 Remove potting boot.

8.0 TEST PROCEDURE

The procedure shall be carried out in the following sequences:

- 8.1 The sample, prepared per Paragraph 6.0, shall be mounted in the horizontal sample holder and positioned within the test chamber by fastening the stripped end of each 7" wire to an electrical connection. Mate and lock the specimen PT06CE electrical connector to the PT07CE Jam Nut Receptacle (prepared per 5.0) on the central connector mount.
- 8.2 The test chamber shall be evacuated to a pressure of 1 Torr and repressurized to the test pressure with oxygen. Allow the chamber to stand for one minute. A leak is indicated if an increase in test chamber pressure is observed after the vacuum pump is closed off from the system. The system shall be brought to atmospheric pressure and the leak corrected before any additional tests are carried out. Repeat the above procedure.
- 8.3 After the test chamber has been stabilized at the test pressure, soak the specimens ten minutes. Apply a current of 40 amperes to the wire. If ignition or considerable degradation is not obtained in one minute, the current shall be increased by 5 amperes (i.e., from 40 to 45 and 45 to 50, etc.) until such time as the wire fails or ignition occurs. If ignition occurs prior to wire failing, reduce current to zero amperes to evaluate self-extinguishing characteristics of potting compound.
- 8.4 Three samples of each potting compound shall be tested. The failure of any one sample to meet the criteria of Paragraph 3.0 shall be cause for rejection of that compound.

9.0 REPORT

The following test data and pertinent information concerning the materials shall be reported:

- 9.1 Name of the material (generic).
- 9.2 Vendor designation and vendor.
- 9.3 Usage, quantity, and surface area in the spacecraft.
- 9.4 Results of each test including combustion phenomena if ignition occurs.
- 9.5 Date of test.
- 9.6 Test number.
- 9.7 Identity of the testing agency or component.
- 9.8 Name of the test coordinator.

FIG. 1 POTTING FLAMMABILITY TEST EQUIPMENT SCHEMATIC

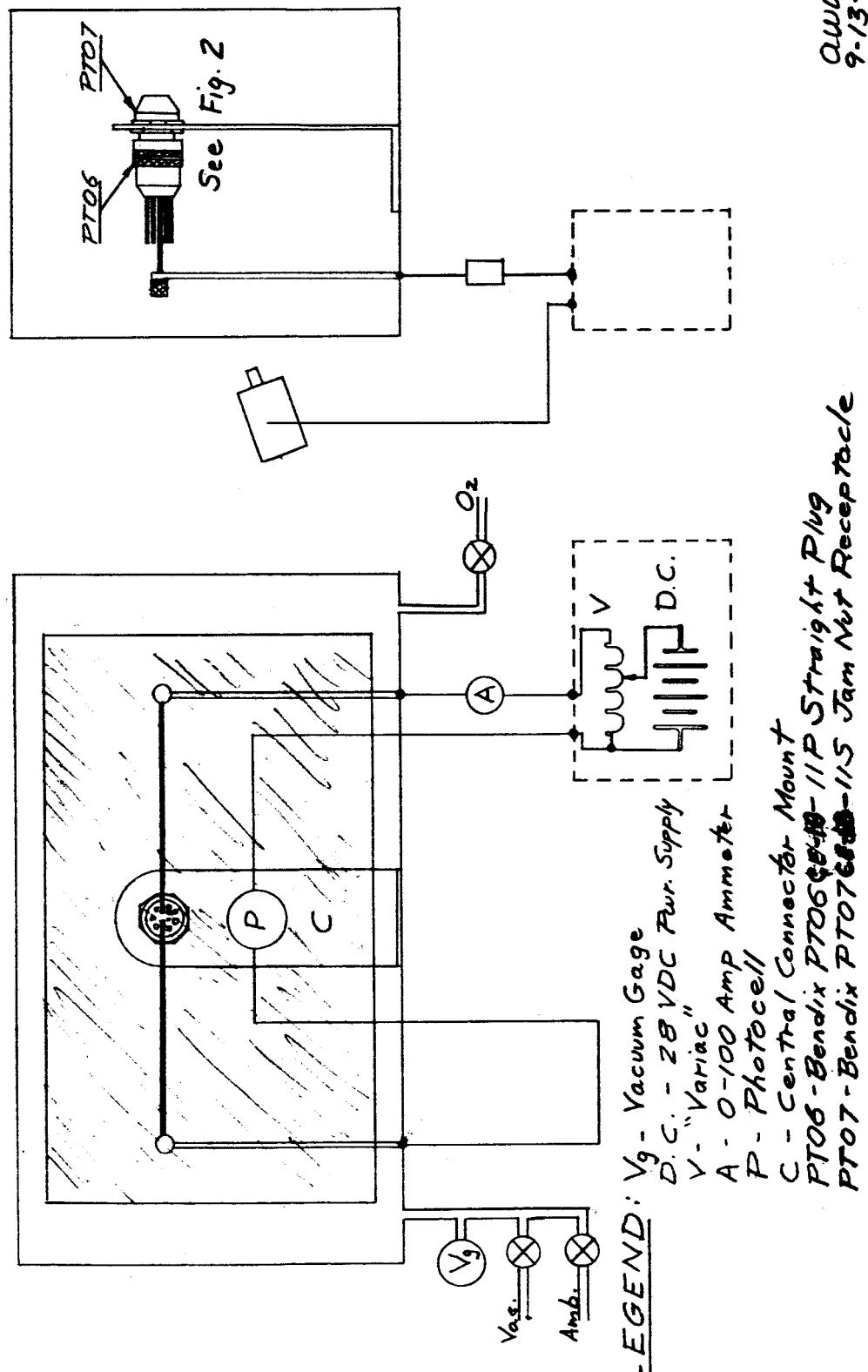
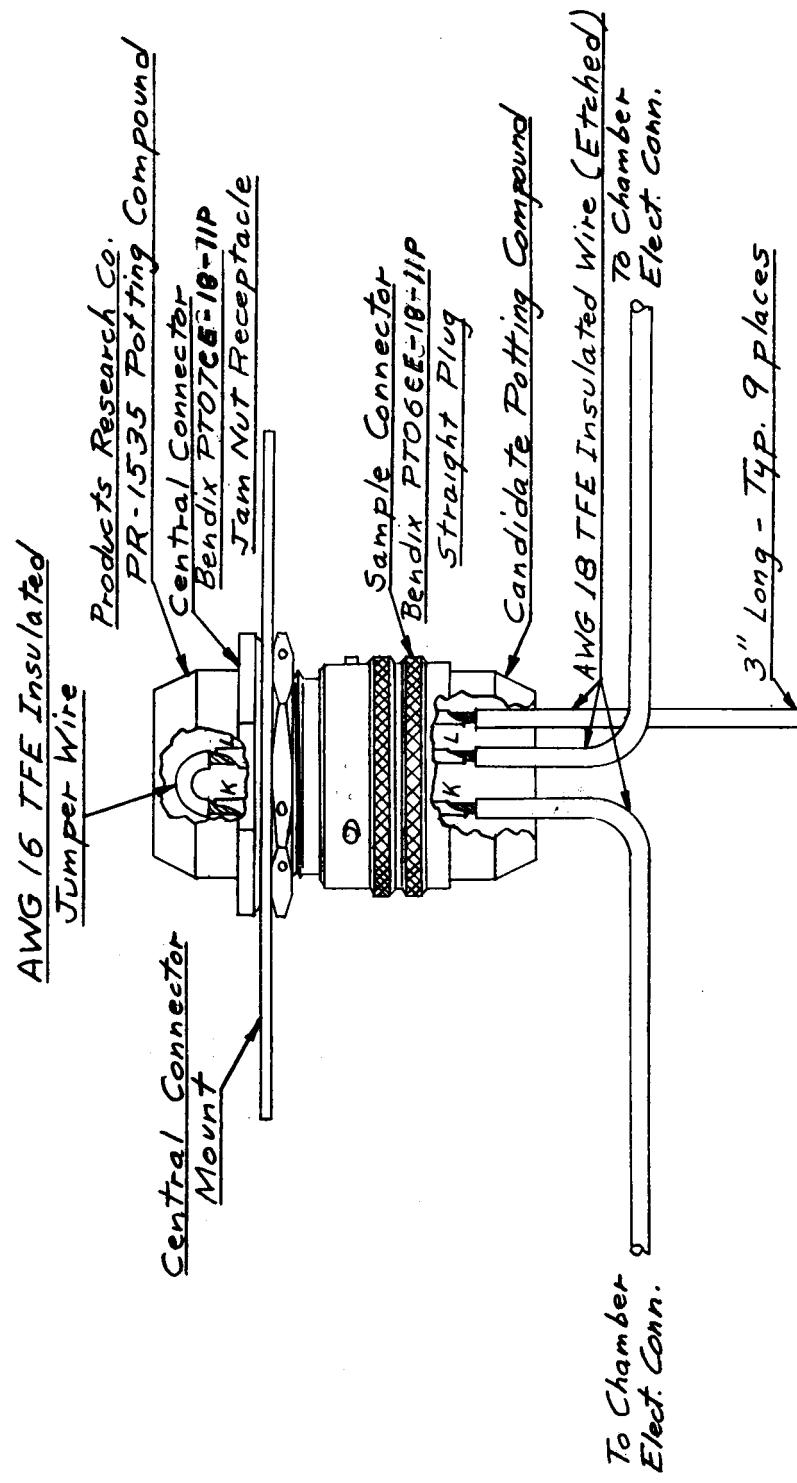


FIG. 2 SAMPLE CONFIGURATION



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TEST NO. 6

ODOR TEST

1.0 PURPOSE

These procedures are designed to eliminate unsuitable materials from use in the habitable area of spacecraft. A material that fails this specification shall not be used.

2.0 ODOR CRITERIA

2.1 Selection of Test Panel for Odor Evaluation - The test conductor shall establish a pool of qualified personnel.

2.1.1 Members of the pool shall be male and each member be capable of detecting seven basic odors from the following solutions:

<u>Primary Odor</u>	<u>Standard Compound</u>	<u>Amount Dilution in Water</u>
Ethereal	1, 2 - dichoroethane	0.4 ml in 500 ml
Camphoraceous	1, 8 - cineole	5 μ l in 500 ml
Musky	15 - hydroxypentadecanoic acid lactone	1 mg in 1,000 ml
Floral	1 - methyl-1-ethyl-2-phenyl propanol-1	0.075 ml in 500 ml
Minty	menthone (dl)	2 μ l in 333 ml
Pungent	formic acid	25 ml of 90 percent solution in 500 ml

Putrid (methyl disulphide) methyl dithiomethane 1 μ l in 10,000 ml

2.1.2 Members of the pool shall be given three odorless solutions along with the seven primary standards for the detection of odor.

2.1.3 The solutions shall be freshly prepared once a month or as needed.

2.1.4 The established pool for odor evaluation shall be requalified every three months.

- 2.1.5 Members of the pool shall not participate on the panel if their sense of smell is affected in any manner.
- 2.1.6 One of the seven basic odors shall be presented to the panel members as a standard for sensing odor prior to evaluation of odors from any sample material.
- 2.1.7 Panel members shall not be permitted to see or know the sample material being evaluated for odor.
- 2.1.8 Odor evaluations on sample materials shall be performed in a suitable room, free from extraneous odors.
- 2.1.9 Odor evaluations shall be performed on every new bottle of oxygen used for the tests.
- 2.2 Odor Evaluation - Each panel member shall evaluate the odor of a sample according to the following scale:

<u>Members Rating</u>	<u>Test Conductor's Rating</u>
Undetectable	0
Barely detectable	1
Easily detectable	2
Objectionable	3
Irritating	4

- 2.2.1 A total score of 20 or less, for the sum of ten odor evaluations of any sample material by the panel members, signifies the material passes the odor test. A total score above 20 at any dilution signifies that the material fails the odor test and is rejected.

3.0 TEST DISCIPLINE

- 3.1 All materials shall be tested for carbon monoxide content on the Kitegawa before being subjected to the odor test in order to prevent possible poisoning of the panel members.
- 3.2 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.

- 3.3 Each test shall be witnessed by the assigned Quality Assurance Inspector and each step appropriately verified and stamped as required by the procedure.
- 3.4 Approval of the test shall be indicated at the end of the test procedure by the signature of the responsible Test Engineer and Quality Assurance Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the Quality Assurance Inspector.

4.0 TEST EQUIPMENT

- 4.1 Test Chamber - The test chamber shall be made of pyrex glass and its internal volume shall be two liters minimum. The test chamber shall have the following:
 - 4.1.1 A gas tight removable cover.
 - 4.1.2 A sampling valve.
 - 4.1.3 A sampling port capable of being sealed with a septum.

A laboratory vacuum dessicator may be utilized as a test chamber.
- 4.2 Oven - The oven shall be capable of providing a constant temperature of $155\pm 5^{\circ}\text{F}$.
- 4.3 Oxygen Supply - The oxygen shall conform to MIL-0-27210, Type 1. Suitable equipment for transferring oxygen to test chamber shall be used.
- 4.4 Sample Transfer Equipment - Glass syringes shall be used for measuring and transferring the sample atmospheres from the test chambers to panel members face mask.
- 4.5 Pressure Gauge - The pressure gauge shall be capable of measuring pressures from 1 Torr. to 760 Torr. to within ± 1 Torr. accuracy.
- 4.6 Olfactometer - The olfactometer shall consist of a mask made of odorless flexible material which can be applied to a panel member's face.
- 4.7 All odor testing equipment shall be nonproducers of odor and carbon monoxide, under test conditions set forth under Section 6.0.

5.0 SAMPLE PREPARATION

- 5.1 Sample materials that have a defined volume or shape shall be cut into strips one eighth inch thick or less with sufficient length and width to make up a 5.00 ± 0.25 gm sample per liter of test chamber volume.
- 5.2 Film and fabric materials shall be tested in their "as purchased" condition,

with sufficient area to make up a $5.00 \pm .025$ gm sample per liter of test chamber volume.

5.3 Primers, coating materials, and adhesives shall be applied to a chemically clean 0.030 inch thick aluminum substrate panel. The coating materials shall be applied in a thickness equivalent to normal practice of use, and shall be post cured according to manufacturing practice. A total quantity of 5.00 ± 0.25 gm sample per liter of test chamber volume shall be used.

5.3.1 The total coated panel area to be tested shall be 300 square centimeters per liter of test chamber volume. The panels may be cut into smaller pieces for convenience of exposure in the test chamber.

5.3.2 The panels may be coated on both sides.

5.4 Liquids such as greases, oils, coolants, etc. shall be contained as samples in aluminum weighing dishes of approximately two inches in diameter. The liquid test samples shall weigh 5.00 ± 0.25 gm per weighing dish and per liter of test chamber volume.

6.0 TEST CONDITIONS

- 6.1 The atmosphere in the test chamber at the start of the test exposure shall be MIL 0-27210, Type 1, oxygen at 260 ± 5 Torr.
- 6.2 The sample materials shall be heated at a temperature of $155 \pm 5^{\circ}\text{F}$ for a test duration of at least 72 hours.
- 6.3 Leak Check - The test system shall not increase in pressure more than one Torr, while remaining at a reduced pressure of one Torr, for a time period of one hour.
- 6.4 Each sample material shall be tested in a separate test chamber.
- 6.5 Carbon monoxide measurements and odor evaluations shall be carried out within three hours of the conclusion of the thermal treatment.
- 6.6 All measuring equipment shall have the proper calibration stickers.

7.0 TEST PROCEDURE

The procedure shall be conducted in the following order.

7.1 Sample materials shall be prepared according to the conditions outlined

under sample preparation in Section 5.0.

- 7.2 After placing sample materials in separate test chambers, the test chamber shall be evacuated to one Torr. or less. The test chambers shall then be pressurized to 260 ± 5 Torr. with MIL 0-27210, Type 1, oxygen.
- 7.3 The test chambers shall be exposed to a temperature of 155 ± 5 °F in an oven for a time period of at least 72 hours, allowing time for initial warm-up.
- 7.4 Following the isothermal exposure, the test chambers shall be removed from the oven and allowed to return to room temperature.
- 7.5 The pressure in the test chambers shall be measured and recorded.
- 7.6 Observation of distillable residues on interior chamber walls shall be made and recorded. A material that deposits a residue on the chamber walls may be cause for rejection.
- 7.7 The test chambers shall be pressurized to one atmosphere with oxygen and a sampling septum installed.
- 7.8 A sample shall be withdrawn and the carbon monoxide content determined by the Kitegawa method. A content in excess of 25 ppm shall preclude odor testing at this time. The odor test shall be resumed if the material evolves less than 25 ppm carbon monoxide.
- 7.9 Odor Test* - Known volumes of sample atmosphere shall be extracted from the test chambers by means of a syringe and diluted with fresh oxygen in the following proportions:
 - 7.9.1 One part of sample atmosphere to 29 parts of oxygen.
 - 7.9.2 One part of sample atmosphere to 9 parts of oxygen.
 - 7.9.3 No dilution, or as drawn from the flask.

8.0 REPORTING

Unless otherwise specified, the following test data and pertinent information concerning the materials shall be reported.

- 8.1 Name of material.
- 8.2 Vendor designation and vendor.
- 8.3 Carbon monoxide content as determined by the Kitegawa.

*A material that fails the criterion at any dilution described in Section 7.9 is unequivocally rejected for spacecraft usage.

- 8.4 Rating numbers determined by the panel members.
- 8.5 Definition of the odor as interpreted by each panel member.
- 8.6 Status of the material as a result of the test.
- 8.7 Name and number of the test procedure.
- 8.8 Date of test.
- 8.9 Identity of the testing agency.
- 8.10 Name of test coordinator.

TEST NO. 7

DETERMINATION OF ORGANIC OFFGASSING PRODUCTS AND CARBON MONOXIDE

1.0 PURPOSE

This procedure establishes the criteria for determining the suitability of nonmetallic materials for use in the space vehicle crew compartment environments, with respect to production of potentially toxic or objectionable materials by outgassing.

2.0 SIGNIFICANCE

The amount, nature and rate at which potentially toxic and objectionable products outgas from crew bay materials must be evaluated and analyzed in order to insure the complete physiological safety and optimum mission performance of the astronauts in manned space vehicles. To be able to attain reasonable certainty that the environment of the spacecraft is toxicologically safe, it is necessary to establish a maximum allowable contaminant level for the spacecraft and maximum levels for individual components.

3.0 CRITERIA OF ACCEPTABILITY

The outgassing concentration of individual major contaminants is related to the effective spacecraft crewbay volume.

- 3.1 A material which does not offgas organic compounds under the test conditions may be used freely in the spacecraft crew bay area.
- 3.2 The maximum allowable level of total organics in the as configured spacecraft shall not exceed 100 ppm at 14.7 psia or 300 ppm at 5.0 psia.
- 3.3 The maximum allowable level of carbon monoxide in the as configured spacecraft shall not exceed 25 ppm at 14.7 psia or 75 ppm at 5.0 psia.

TEST DISCIPLINE

- 3.4 Each test shall be directed by the cognizant test engineer or his appointed alternate.
- 3.5 Each test shall have Quality Assurance coverage and each step of the procedure or technique used shall be appropriately verified and stamped as required.

3.6 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible test engineer and QA inspector. Entries transcribed to the test data sheets will be also verified by the test engineer and the QA inspector.

4.0 TEST EQUIPMENT

4.1 Test Chamber - The test chamber shall have a minimum main chamber volume of two liters. It shall have a configuration and be fabricated of materials which allow ready cleaning. Nonmetals, other than glass composing the chamber and gas handling system, shall be proven to give off negligible gaseous products under the test conditions by running blank or control tests. Provision shall be made to prevent condensation or trapping of gaseous products other than in the room temperature condenser. A thermometer or thermocouple and pressure gauge for temperature and pressure determination, respectively, shall be included in the test chamber. The chamber shall be connected to a series of traps and inlet tubes to permit evacuation, pressure readings, oxygen introduction, helium flow for trapping purposes, and sample withdrawal for quantification of volatile outgassing components. An alternate system would be to connect the chamber directly to a gas chromatograph and/or mass spectrometer for the gas analyses.

4.2 Heating Source - The oven shall maintain constant temperature control over the test chamber to $\pm 5^{\circ}\text{F}$. The temperature during sample exposures shall be recorded.

4.3 Vacuum Pump - The vacuum pump shall be capable of producing a vacuum less than one Torr. and little or no system contaminants.

4.4 Analytical Equipment - The analytical equipment shall consist of the following and any other instruments which the tester desires to use to completely and efficiently evaluate offgassing products. These equipment items shall be identified.

4.4.1 Gas Chromatograph System - Recorder, thermal conductivity and hydrogen flame ionization detectors. The columns shall have the capability of separating light organic and inorganic gases, organic sulfides and mercaptans, halogenated hydrocarbons, representative aliphatic and aromatic hydrocarbons including aldehydes, ketones, alcohols, and esters. The hydrogen flame ionization detector has greater sensitivity to organic materials. Conversely, because of the flame ionization detector's lack of sensitivity to the other compounds listed above, the thermal conductivity detector is used for their detection.

- 4.4.2 Recording Infrared Spectrophotometer with accessories capable of analyzing three microliters (μ l) or less of liquid.
- 4.4.3 Mass Spectrometer
- 4.4.4 Trapping System suitable for trapping and transfer of microliter quantities of liquid from the gas chromatograph to the infrared spectrophotometer or mass spectrometer. A direct G.C.-mass spectrometer connection can replace the trapping system if desired.
- 4.4.5 Gas Sampling System suitable for transfer of measured volumes of gas samples from the test chamber to the gas chromatograph.
- 4.4.6 Calibration Gas Samples as required to quantify detector sensitivity and readout. These may be bought from a manufacturer or made up in the laboratory.

5.0 PREPARATION OF TEST EQUIPMENT

- 5.1 Prior to analysis, all glassware must be washed in a hot detergent solution, rinsed in distilled water, and dried at 400°F for 1/2 hour. Periodically, a completely clean system shall be checked for contamination by withdrawal of a sample for analysis by gas chromatography.
- 5.2 The test system shall be free of leaks. Checks for leaks shall be made before each test.

6.0 TEST SPECIMENS

- 6.1 Coatings, finishes, primers, adhesives, solid film lubricants, etc. shall be applied to a chemically cleaned aluminum substrate of suitable thickness and in a coating thickness equal to production practice. The aluminum surface shall be approximately 300 square centimeters per liter of test chamber volume and coated on one or both sides depending on the chamber volume.
- 6.2 Films, fabrics, plastics, etc. other than foams shall be exposed when possible to an area of sixteen square inches and an approximate thickness of 1/32 inch.
- 6.3 Foams shall be 1/2 inch thick and approximately sixteen square inches in area.
- 6.4 Electrical insulation specimens, where possible, shall be in four inch lengths or quantity equivalent to an exposed surface area of sixteen square inches.
- 6.5 Specimens shall be brush cleaned.
- 6.6 Fluids, greases, and non-conformable materials shall be placed in chemically cleaned, tared, aluminum containers for analysis.

- 6.7 Specimens shall be stored, when required, in an air tight container at ambient temperature and pressure.
- 6.8 All samples shall be weighed prior to and after exposure and the weight change shall be determined to an accuracy of ± 0.1 percent. All specimens except thin film material should weigh approximately ten grams and should be weighed to the nearest 0.1 milligram prior to the test. The thin film material shall be weighted to the nearest 0.1 milligram prior to the test and the weight recorded.

7.0 PROCEDURE

- 7.1 Purge the test chamber with MIL 0-27240, Type 1, oxygen until the minimum oxygen concentration is 95 percent and begin heating. Bring the chamber to $155^\circ \pm 5^\circ\text{F}$ and adjust the chamber pressure to 5.0 ± 0.1 psia.
- 7.2 After twenty-four hours, check the oxygen for contaminants with the gas chromatograph. Proceed to 7.3 if the total contamination is less than 10.0 ppm by volume. If the contamination exceeds the above values, the test chamber shall be flushed and rechecked after an additional twenty-four hours. This procedure shall be continued until the contamination is below the specific level of ten ppm. If the gassing contamination continues indefinitely, revision of the test chamber is indicated.
- 7.3 Place a clean weighed specimen prepared per Section 6.0 in the test chamber.
 - 7.3.1 The chamber shall be evacuated through a liquid nitrogen trap to below one Torr. The chamber shall then be closed off, heat applied, and oxygen filtered through a 5X Molecular Sieve bled into the chamber. The conditions of exposure are held at 5.0 ± 0.1 psia of 95 percent pure oxygen and $155^\circ \pm 5^\circ\text{F}$ for a total period of 72 hours if required. The offgassing rate shall be determined by drawing off samples 8, 24, 48 and 72 hrs, then analyzing and plotting the results on a graph and evaluating the resultant curve. Condensates in the cold trap shall be recovered as gas and likewise analyzed and plotted.

The gases are then analyzed for the following:

- 7.3.1.1 Total organics expressed as methane equivalents.
- 7.3.1.2 Complete analysis of individual outgassing components above 10 ppm.
- 7.3.1.3 Quantity of carbon monoxide evolved.

Following the designated exposure period, the chamber is brought to room temperature. The test specimen is removed from the chamber and weighed after the final gas samples are taken.

- 7.4 Determination of Total Organics - The gas chromatograph with the pertinent columns, Beckman Total Hydrocarbon Analyzer or an equivalent instrument may be used for the determination.
- 7.5 Identification of Outgassing Components Exceeding 10 ppm - When specified by the test monitor, constituents exceeding 10 ppm in the spacecraft atmosphere shall be positively identified.
- 7.6 Quantification of Individual Outgassing Components - Gas sample standards of each components isolated and identified in Section 7.5 are required for the quantification. The standards may be bought commercially or made up in the laboratory.
- 7.7 Determination of Carbon Monoxide - The carbon monoxide content of the evolved gases shall be determined under Sections 7.4, 7.5, and 7.6 by utilizing an appropriate separation and analytical technique.

8.0 REPORTING DATA

- 8.1 The following test data and pertinent information concerning material tested shall be reported:

- 8.1.1 Name of the material (generic)
- 8.1.2 Vendor designation and vendor
- 8.1.3 Usage, quantity, and surface area in the spacecraft
- 8.1.4 Results of tests
 - 8.1.4.1 Carbon monoxide ppm per gram based both on the test chamber volume and on the spacecraft volume.
 - 8.1.4.2 Total organics ppm per gram based both on the test chamber volume and on the spacecraft volume as methane equivalents.
 - 8.1.4.3 Identity of organics greater than 10 ppm.
 - 8.1.4.4 Weight loss, percent.
 - 8.1.4.5 Rate of outgassing for total organics in ppm for 8, 24, 48 & 72 hours.
 - 8.1.4.6 Amount and type of condensate in traps.
- 8.1.5 Disposition or status, dimensions, and size of sample material
- 8.1.6 Name and number of test procedure
- 8.1.7 Date of test
- 8.1.8 Test number
- 8.1.9 Identity of the testing agency or component
- 8.1.10 Name of test coordinator

TEST NO. 8
TEST FOR MATERIALS IN VENTED CONTAINERS

1.0 PURPOSE

This procedure describes the method for determining the combustion characteristics and effects on adjacent installations of a fire in a vented container.

2.0 TEST CONDITIONS - PRESSURES

This test shall be performed for category "F" materials configured in the container as if under flight conditions. If the CM assembly fails to meet the requirements when tested at 16.5 psia oxygen, it shall be retested at 6.2 psia and the results reported.

<u>Categories</u>	<u>Module</u>	<u>PSIA (oxygen)</u>
F	CM	16.5 and 6.2
F	LM	6.2

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

In the event of an internal fire, the container:

- 4.1 Shall not rupture and thus permit the scattering of flaming contents about the cabin.

- 4.2 Shall not permit flames to emit through vents and propagate to nearby nonmetallic materials.
- 4.3 Shall not emit heat to the extent that the autogenous ignition temperatures of adjacent nonmetallic materials are reached.
- 4.4 Shall not permit smoke and noxious fumes to escape into the cabin to the extent that the astronauts may be adversely affected.
- 4.5 Shall not emit gases and heat to the extent that a rise in cabin pressure shall exceed 2.5 psia.

5.0 TEST EQUIPMENT

- 5.1 The test chamber shall be the mock-up of the command module complete with the inlets for the required temperature and pressure measuring devices, vacuum, oxygen, air and ignition wires.
- 5.2 Propagation Rate Indicators
 - 5.2.1 Temperature Monitoring - Thermocouple readings may be monitored by multiple point recorders or any equally accurate method. The desired accuracy is $\pm 10^{\circ}\text{F}$. The thermocouples shall be placed inside the box immediately proximate to but not touching the ignition source and approximately at the center of the top, bottom and four sides of the interior. Thermocouples shall be placed on the exterior of the container against the top, bottom and four sides and one-half inch above and one-quarter inch lateral to each vent or vent area.
 - 5.2.2 Motion pictures may be taken of the test.
 - 5.2.3 Ignition Source - Ignition of the materials in the container shall be accomplished by utilizing a nichrome wire wrapped around its center portion with a 1" x 2" piece of facial type tissue and placing it in contact with the item selected as the prime source of ignition. The power supply to the wire shall provide sufficient voltage by means of a variable transformer to ignite the material.

6.0 SAMPLE PREPARATION

- 6.1 The container shall be the same size, geometry and material as the flight container.

- 6.2 The equipment to be tested shall be the same, with regard to the nonmetallics, as the flight equipment. Electronic parts and similar expensive metallic or ceramic entities may be simulated.
- 6.3 The equipment shall be packaged and positioned in the container in the same manner as in the flight article.

7.0 PRETEST PROCEDURE

- 7.1 Verify that all test equipment is in current calibration.
- 7.2 Verify that the container is the required item.
- 7.3 Witness attachment of thermocouples in the designated areas.
- 7.4 Observe the packaging and positioning of the materials and equipment.
- 7.5 Verify the oxygen certification (MIL-0-27210, Type 1)
- 7.6 Verify material identification as one of the following:
 - 7.6.1 Manufacturers' Certification
 - 7.6.2 NASA Certification
 - 7.6.3 Contractor Certification
 - 7.6.4 Definite identification not available.
- 7.7 Position or observe the positioning of the container in the mock-up in the same position in which the flight article would be situated.
- 7.8 Verify the placing of the ignition wire in the designated spot in the container.
- 7.9 Observe that the ignition wire and thermocouples and other instrument leads are hooked up to the pertinent outlets.

8.0 TEST PROCEDURE

- 8.1 The chamber shall be evacuated to a maximum of 5 Torr and sampling sustained for 5 minutes.
- 8.2 Isolate the chamber and monitor the pressure for one minute in order to check for leaks. A rise of one torr during that time indicates a leak which must be rectified before going to the next step.

- 8.3 Pressurize the chamber to 16.5 ± 0.1 psia or 6.2 ± 0.1 psia for the LM, with oxygen.
- 8.4 Analyze the chamber to verify that a minimum of 95% oxygen exists.
- 8.5 After the chamber has stabilized at the proper psia, permit the container to equilibrate for 15 minutes.
- 8.6 Verify that the chamber pressure is at test pressure ± 0.1 psia and isolate the chamber.
- 8.7 Start all applicable instruments and the motion picture camera, if used.
- 8.8 Apply current to the ignition wire.
- 8.9 Observe all phenomena occurring in and around the container.
- 8.10 Record any pressure rise and the final pressure in the chamber.
- 8.11 Remove the container and examine it and the contents and record all observations.

9.0 REPORTING

- 9.1 Identification by generic name of all the materials tested in the container.
- 9.2 Vendor and vendor designation.
- 9.3 Construction and source of container.
- 9.4 Combustion phenomena
 - 9.4.1 Presence or absence of flame or ignition failure.
 - 9.4.2 Color of flame, if present.
 - 9.4.3 Evolution of smoke from the box.
 - 9.4.4 Evolution of flame from the vents.
 - 9.4.5 Length of time flame persisted.
 - 9.4.6 Length of time smoke evolved.

- 9.5 Rate of pressure rise and final pressure.
- 9.6 Status, dimension and size of sample materials.
- 9.7 Condition of container after the test.
- 9.8 Condition of contents of the container after the test.
- 9.9 Name and number of test procedure.
- 9.10 Date of test.
- 9.11 Test number.
- 9.12 Identity of the testing agency or component.
- 9.13 Name of test coordinator.

TEST NO. 9
ELECTRICAL OVERLOAD TEST FOR HERMETICALLY
SEALED CONTAINERS

1.0 PURPOSE

This procedure describes the method for testing materials packaged in hermetically sealed containers and the ability of the containers to withstand, without rupturing, the effects of a fire initiated within the container as a result of an electrical overload.

2.0 TEST CONDITIONS

This test shall be performed for category "E" materials configured in the container containing an internal ignition source in the same manner as the flight item. "Worst case" configuration of materials, i.e., a concentration of nonmetallic materials in close proximity and adjacent to a high voltage or current source, shall be utilized in the test.

The test shall be performed under actual flight conditions. If the CM assembly fails to meet the requirements when tested at 16.5 psia oxygen, it shall be retested at 6.2 psia and the results reported. Following are the pressures at which the containers must be tested according to the module in which they are used.

<u>Categories</u>	<u>Module</u>	<u>PSIA (oxygen)</u>
E	CM	16.5 and 6.2
E	LM	6.2

3.0 TEST DISCIPLINE

- 3.1** Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2** Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3** Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the test engineer and the QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

The hermetically sealed container shall not rupture or exceed the design leakage requirements as a result of the test.

5.0 TEST EQUIPMENT

5.1 The test chamber shall be large enough to accomodate the largest container to be subjected to the test with enough additional volume to allow observation of the extent of scatter in case of rupture and the attendant rise in pressure and temperature in the chamber.

The chamber shall be complete with inlets for the required temperature and pressure measuring devices, vacuum, oxygen, air and ignition wires.

5.2 Thermal Characteristics Indicators

5.2.1 Temperature Monitoring - Thermocouple readings may be monitored by multiple point recorders or any equally accurate method. The desired accuracy is $\pm 10^{\circ}\text{F}$. The thermocouples shall be placed inside the container spaced one inch apart along and in contact with the conductor selected for overloading. Additional thermocouples shall be placed approximately at the center of the top, bottom and four sides of the interior. Thermocouples shall be placed on the exterior of the container against the top, bottom and four sides, approximately in the middle of each panel.

5.2.2 A motion picture camera may be set up to film the phenomena in case the container distorts or ruptures.

5.2.3 Ignition Source - Ignition of the materials in the container shall be accomplished by utilizing a power supply which is capable of providing a large steady current through one wire of the conductor bundle so that a very high temperature will be achieved quickly. The power source must be capable of supplying a current 10 percent above the nominal fusion current for that gauge wire being tested. See Test No. 4, Section 4.5 for the nominal fusion current for wire gauge numbers 28 through 12.

6.0 SAMPLE PREPARATION

6.1 The container shall be the same size, geometry and material as the flight container.

- 6.2 The equipment to be tested shall be the same, with regard to the non-metallics, as the flight equipment. Costly electronic parts and similar expensive metallic or ceramic entities shall be merely simulated in geometry and mass using the same basic materials.
- 6.3 The equipment shall be packaged and positioned in the container in the same manner as in the flight article. Thermocouples shall be exited through bushings or any adequate hermetically sealable means.

7.0 PRETEST PROCEDURE

- 7.1 Verify that all test equipment is in current calibration.
- 7.2 Verify that the container is that which is required.
- 7.3 Observe that the thermocouples are attached in the designated areas.
- 7.4 Verify the packaging and positioning of the materials and equipment.
- 7.5 Verify the oxygen certification (MIL-0-27210 Type I)
- 7.6 Verify materials identification as one of the following:
 - 7.6.1 Manufacturers' Certification
 - 7.6.2 NASA Certification
 - 7.6.3 Contractor Certification
 - 7.6.4 Definite identification not available.
- 7.7 Position or observe the positioning of the container in the chamber.
- 7.8 Observe that the thermocouples and other instrument leads are hooked up to the pertinent outlets.
- 7.9 Verify that the conductor to be overloaded is hooked up to the proper, high current power supply.
- 7.10 Observe that the chamber is filled with its functional atmosphere, closed and made secure for the test.

8.0 TEST PROCEDURE

- 8.1 The chamber shall be evacuated to a maximum of 5 torr and pumping sustained for 5 minutes.
- 8.2 Isolate the chamber and monitor the pressure for one minute in order to check for leaks. A rise of one torr during that time indicates a leak which must be repaired before going to the next step.
- 8.3 Pressurize the chamber to 16.5 psia or 6.2 for the LM, with oxygen.
- 8.4 Verify that the chamber is at test pressure ± 0.1 psia and isolate the chamber.
- 8.5 Start all applicable instruments.
- 8.6 Apply the current to the selected conductor starting at the amperage shown for that size conductor in the table in Section 4.5 of Test No. 4. If no effect is shown, the current shall be increased in 5 amps steps at one minute intervals until the wire fails or ignition occurs.

Observe and record whether ignition occurs or wire failed.
- 8.7 If ignition occurs, record all phenomena including temperature rises inside and outside the chamber and any measurable pressure increases.
- 8.8 Remove the container and examine it and the contents and record all observations.

9.0 REPORTING

- 9.1 Identify by generic name all the nonmetallic materials tested in the container.
- 9.2 Vendor's designation and vendor.
- 9.3 Construction and source of container.
- 9.4 Observations
 - 9.4.1 Presence or absence of ignition or fusion of the conductor.
 - 9.4.2 Temperature changes inside the container and those measured on the outer walls of the container.

- 9.4.3 Pressure rise inside the container during the test.
- 9.4.4 Phenomena associated with rupture of container, if rupture occurred:
 - 9.4.4.1 Presence or absence of flame and smoke.
 - 9.4.4.2 Extent of scatter of burning or smoking materials.
 - 9.4.4.3 Part of container where rupture occurred.
 - 9.4.4.4 Pressure and temperature rise in chamber.
- 9.5 Dimension, size and status of sample materials.
- 9.6 Condition of container after the test.
- 9.7 Condition of the contents after the test.
- 9.8 Name and number of the test procedure.
- 9.9 Date of test.
- 9.10 Test number.

TEST NO. 10
GUIDELINES FOR SIMULATED PANEL AND ASSEMBLY
FLAMMABILITY TESTS

1.0 PURPOSE

This procedure describes the method for determining the combustion characteristics, particularly the propagation rate, of a number of different size and areas of nonmetallic materials of similar or different types which make up a functional assembly, subsystem or system in the spacecraft. The sizes and areas of the materials are such that the materials fall into Category B of the Nonmetallic Materials Selection Guidelines and Test Requirements.

(The procedure is under development. Guidelines for establishing the parameters of the test follow.)

2.0 OBJECTIVE

The objective of fire tests on assemblies is to determine the behavior of the assembly under the particular test conditions, and at the same time to furnish data which can be basis for the formulation of generalized fire models for the simulation of many different fire situations. In this fashion, a limited number of well instrumented fire tests on assemblies can lead to the ability to predict fire hazard situations for enumerable assemblies under a variety of conditions. The generation of this type of ability would simplify the problem of designing and assessing the reliability of manned spacecraft to a large extent. Each test, then, should be designed to be complete not only of itself, but to be a part of the larger picture of a model of the fire situation of the entire spacecraft.

3.0 TEST GUIDELINES

In general, two modes of ignition must be considered:

1. Ignition from an internal source, i.e., wire overload or short circuit.
2. Ignition from an external source, i.e., a flame or fire propagated from another nearby burning entity.

Naturally, the effects of burning of the assembly must be considered in light of its influence on the spacecraft external to the assembly, as well as the internal effects of the fire on the assembly.

The information desired as a result of the fire tests is:

- A. Temperature distribution within the assembly.
- B. Initial point of ignition.
- C. Total heat generated.
- D. Increase in pressure in the chamber.
- E. Flame propagation paths within the assembly.
- F. Heat transport paths away from the assembly.
- G. Plume characteristics (temperature, size, shape).
- H. Characteristics of burning (sparks, burning rate, drips, etc.)
- I. Quantity and composition of combustion product gases.

The steps in the overall test program to obtain this information are as follows:

- A. Define or select the test assembly. This should be a portion of the spacecraft which is a meaningful and convenient package for fire hazard testing and analysis. The portion selected shall be that definable area in which a potential fire will be contained (See Item B, Paragraph III, ASPO-RQTD-D67-5A).
- B. Perform a fire hazard analysis on the test assembly.
 - 1. Material inventory
 - 2. Configuration analysis
 - 3. Thermal analysis
- C. Design the fire test to produce the required information. The experimental design will be based on the fire hazard analysis.
- D. Write detailed test procedure.
- E. Conduct the test.
- F. Report and evaluate the results.
- G. Make recommendations.

2.2 Specific Guidelines

A. Test Conditions

- 1. Configuration and materials of the test assembly will duplicate exactly the item to be simulated.
- 2. Test assembly (combustion element) will be oriented in the test chamber as it will be in the operational spacecraft at launch.

3. The test chamber volume will be sufficient to accomodate the largest assembly to be tested with adequate space around the assembly to allow observation of the extent of projection of burning pieces due to sputtering and expansion of gases internal to the burning mass.
4. For checking out an assembly mounted in a completed space-craft, the test assembly must be identical to the one in the spacecraft. However, to demonstrate the feasibility of over-coating the flight assembly with a fire retardant compound and, thereby, render a retrofit unnecessary, the test assembly must be coated with the same fire retardant material and then tested for compliance with the flammability requirements.
5. The test assembly will be operational prior to and during the test and will be tested for operation after the test.
 - a. The worst case, mode of operation, determined by the fire hazard analysis, will be selected.
 - b. The test assembly will be in operation prior to the test until steady state conditions are reached.
6. For assemblies containing substitute materials or new designs, the test configuration need not be operational unless economically feasible. Only the nonmetallic materials shall be configured as in the flight article. Expensive components may be simulated, but the basic material, geometry and mass of the components must be the same as in the flight assembly.
7. All thermal interfaces will be simulated.
 - a. Cold plates
 - b. Structure
 - c. Convection due to the cabin fan
 - d. Cabin radiation characteristics
8. Environment
 - a. Test chamber atmosphere temperature will be 75°F at start of test.

b. Pressure of pure O₂.

- (1) LM - 6.2 psia
- (2) CM - 16.5 psia

c. O₂ added to chamber at normal spacecraft addition rate (during testing).

9. Instrumentation

Instrumentation requirements for each test assembly will be determined on an individual basis as part of the overall combustion hazard analysis. Typical requirements will include:

a. High speed color movies of each test so that at least the following combustion characteristics may be recorded.

- (1) Flame geometry
- (2) Flame propagation on and within assembly
- (3) Burning rate
- (4) Combustion characteristics, i.e., sputtering, sparking, dripping, etc.
- (5) Time base

Complete identification of each test will be recorded on the film before and after the test.

- (a) Spacecraft
- (b) Assembly
- (c) Date
- (d) Test personnel
- (e) Location
- (f) Referenced test procedure

b. Temperature Measurements

Temperatures will be continuously recorded during testing.

- (1) Temperature measuring and recording equipment will have response times equal to or greater than the rate of change of the location being measured as predicted by thermal calculations.

- (2) Locations of temperature measurement devices will depend on the configuration of the assembly being tested. These should provide:
 - (a) A complete temperature profile of the assembly
 - (b) Location of ignition sites
- (3) Measure ambient gas temperature.
- (4) Measure chamber wall temperature.

b. Heat Flux Measurements

Measure radiant and total heat flux in all directions from the panel by the use of fast response calorimeters such as asymptotic types or equivalent.

c. Pressure Measurement

Continuously measure and record the chamber gas pressure.

d. Determine quantity and composition of combustion gas generated.

Suggest GC, mass spectrometry methods.

B. Procedure

1. Test procedures or plans will be written and documented prior to each test.
2. Each test procedure will receive the approval of cognizant NASA/MSC personnel prior to test.
3. Ignition

Ignition will be accomplished at appropriate sites (determined by combustion hazard analysis) and will simulate the following fire conditions depending on most likely ignition means determined by the hazard analysis.

- a. Momentary heat source
- b. Ignition source with continuous heat input (internal)
- c. External heat input

A rationale justifying selection of ignition point and means will accompany each test report.

C. Instrument Calibration

1. Standards

Secondary reference standards will be certified against primary reference standards maintained by the NBS. The type of standard and frequency of calibration shall be approved by the RASPO section.

2. Calibration Frequency

Test chamber control and indicating instrumentation shall normally be calibrated monthly against secondary standards. Test item measuring instrumentation shall be calibrated against secondary standards prior to any test.

3. Invalidations

Modification, repairs or changes shall invalidate the previous calibration and the instrumentation shall be recalibrated prior to reuse.

D. Post Test Inspection

The following comparable before and after test data will be obtained.

1. Color still photographs
2. Weight of individual materials
3. Operational characteristics
4. Visual inspection

3.0 TEST REVIEW AND APPROVAL

Specific test plans prepared for panel and assembly tests will be submitted by contractor and GFE organizations for MSC review, prior to the initiation of testing. The responsible test organization should commence test article configuration and instrumentation immediately. If MSC response is not received in 10 working days, test programs may be initiated and test plan approval assured.

TEST NO. 11
GUIDELINES FOR SIMULATED CREW BAY CONFIGURATION
FLAMMABILITY VERIFICATION TEST

1.0 PURPOSE

This procedure will utilize panels and assemblies of "worst case" materials configured in a mock-up vehicle in the same manner as they would be in a flight vehicle for the purpose of determining the propagation and combustion phenomena and behavior of the materials in the event a fire started in the spacecraft.

2.0 TEST CONDITIONS

This test shall be performed on all Category B materials. If any material fails to meet the requirements when tested at 16.5 psia, oxygen, it shall be retested at 6.2 psia and the results reported.

2.1 Test Pressures

<u>Categories</u>	<u>Module</u>	<u>PSIA (Oxygen)</u>
B	CM	16.5 & 6.2
B	LM	6.2

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and QA Inspector.
- 3.4 The test set-up shall simulate the ventilation condition available to the material under test when in the flight condition.
- 3.5 The total volume of the test chamber shall be identical to that of the applicable crew compartment.

4.0 TEST CONFIGURATION

- 4.1 The equipment tested shall be identical to the flight article except where the assembly contains expensive parts or instruments. In those cases, only the nonmetallic materials shall be identical. The parts can be mockups of the same mass and geometry as the flight part or instrument.
- 4.2 All potential propagation paths shall be simulated.
- 4.3 Geometric and spatial proximity to spacecraft panels and enclosures shall be simulated.

5.0 IGNITION SOURCE

- 5.1 A standard hot wire ignition source shall be used. It shall be wrapped with a 1"x2" piece of facial type or standard GSA toilet type tissue paper.
- 5.2 The source shall be placed in contact with the most combustible material in the assembly.
- 5.3 Sufficient energy shall be supplied to the wire to ignite the tissue and the test configuration or to assure that it will not ignite in actual service.

6.0 TEST SELECTION CRITERIA

Tests of "worst case" configurations of each generic class of equipment shall be conducted to verify acceptability.

7.0 TEST EQUIPMENT

The tests shall be performed at MSC utilizing a full scale Apollo mock-up vehicle.

8.0 PRE-TEST AND PROCEDURES

The pre-test procedures will describe, step by step, the preparation of the chamber and the assembly and configuration of the materials within the vehicle.

9.0 TEST PROCEDURES

The test procedures will describe, step by step, the final instrumentation of the test items and the chamber, the performance of the test and the post test disassembly of the test panels for observation.

10.0 REPORTING

Materials identification, test definition and all observations and data analyses shall be reported.

SUPPLEMENTARY TEST NO. A
COMBINED THERMOGRAVIMETRIC ANALYSIS (TGA)
AND SPARK IGNITION TEST

1.0 PURPOSE

This test is designed to investigate combined weight loss and spark ignition characteristics of materials which may be used in the habitable areas of manned spacecraft.

2.0 TEST DISCIPLINE

- 2.1** Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 2.2** Each test shall have Quality Assurance Coverage and each step appropriately verified and stamped as required by the procedure.
- 2.3** Approval of the test conduct shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

3.0 DEFINITIONS

- 3.1** **Flash Point** - The flash point is the lowest temperature at which a material will give off, at or near its surface, flammable vapor, which when mixed with oxygen or air in the environment and exposed to an ignition source will provide a nonself-sustaining flash or flame. This will be indicated in this method by a discernible rise from the base line on the recorder using a photoelectric cell as a sensor, focused on the sample tube.
- 3.2** **Fire Point** - The fire point is the lowest temperature of which the mixture of vapors from the surface of a material and oxygen or other gases continue to burn after ignition. The fire point will be indicated in this method by a discernible sustained rise from the base line on the recorder using a photoelectric cell as a sensor, focused on the sample tube.

4.0 TEST EQUIPMENT

4.1 Thermogravimetric Analyzer (TGA)

The sample chamber shall be constructed of glass or quartz. If chamber temperatures exceed 1000 degrees F a quartz chamber shall be used.

The TGA shall have separation flanges on the TGA column assembly to provide a means of entry. Protection shall be provided against the possibility of operator injury in the event of explosive rupture.

4.2 An electric circuit and apparatus equivalent to that described in Appendix A shall be provided to furnish timed electric power to a spark mechanism within the chamber.

4.2.1 The minimum energy supplied by the spark source shall be at least 30 millijoules.

4.2.2 The spark electrodes shall be made of platinum or any other suitable materials which will not influence the combustion process.

4.2.3 The electrodes shall have a .004 inch radius tip. The spark gap shall be 1/8 inch. The electrodes shall be held in one fixed position during the testing by a suitable fixture and shall be placed 1/4 of an inch above the material at the start of the test.

4.3 Oxygen Supply - The oxygen shall be commercially available oxygen conforming to specification MIL 0-27210, Type 1. Suitable equipment for transferring oxygen to the test chamber shall be provided.

4.4 Pressure Gauge - A pressure gauge capable of measuring pressures from (0 to 760 mm) psia with an accuracy of ± 1 mm psia, or a pressure transducer and recorder with comparable capability shall be used.

4.5 Oxygen or Gas Systems - The oxygen system of the sample chamber shall consist of any suitable tubing to safely conduct oxygen into the sample chamber through this tubing and exhausted directly from the chamber to a vented hood. The gas flow, chamber pressure and composition of gas will vary from test to test. These parameters will be specified on the Test Preparation Sheet (TPS) by the cognizant NASA engineer and noted in the Test Procedure sheets.

4.6 Recorders - Two recorders shall be used. The first recorder shall plot the temperature of the sample chamber on its X-axis. The same recorder shall plot the corresponding weight loss of the material on the Y-axis. The second recorder plots the temperature of the sample chamber on its X-axis exactly as the temperature scale on the first X-axis. The Y-axis of the second recorder plots any ignition light sensed by the photoelectric cell(s).

- 4.7 Photoelectric Cells - A photoelectric cell(s) capable of sensing light over the entire visible spectrum shall be provided and focused on the sample. This photoelectric cell(s) shall be jacketed by a water jacket to reduce any effects of heat from furnace on the photocell.
- 4.8 Thermocouple - Chromel-Alumel, Type K or equal shall be provided for insertion in the thermocouple well of the TGA.
- 4.9 Specimen Holder - Specimens shall be placed in either an open glass grid, a metal platform, or a shallow crucible.
- 4.10 Flow Meter - A flow meter capable of measuring flow rates 10cc/min to 5 liters/min shall be used. It shall be capable of mixing gases in proper proportions if multiple gas atmospheres are used.

5.0 SAMPLE PREPARATION

- 5.1 Bulk materials shall be in the form of circular discs.
- 5.2 Liquids shall be placed in a crucible. The weight of the crucible shall be tared so that it shall not be a recorded weight.
- 5.3 Powders shall be measured in their "as received" form.
- 5.4 Electrical wire insulation shall be stripped from the metal core. The form of sample will be coiled insulation.
- 5.5 Weight - The specimen weight shall be $250\text{mg} \pm 10$.
- 5.6 Materials not suited to testing in the forms described above shall be tested in special forms. In any event, the specimen weight shall not exceed 0.5 grams and the specimen configuration shall be recorded.

6.0 PRETEST PROCEDURE

- 6.1 Verify that all test equipment is in current calibration.
- 6.2 The sample chamber shall be thoroughly cleaned before each test.
- 6.3 Verify gas certification per applicable specification. Contaminants shall be less than .5 percent.
- 6.4 Verify material identification by one of the following:
 - 6.4.1 Manufacturers Certification
 - 6.4.2 NASA Certification
 - 6.4.3 Contractor Certification
 - 6.4.4 Definite identification not available

- 6.5 Prepare samples per paragraphs 5.1, 5.2, 5.3, 5.4, or 5.6.
- 6.6 If irregularly shaped samples are tested describe the shapes.
- 6.7 Clean samples by brushing or by flowing an inert gas over them to remove loose surface contamination.
- 6.8 Weigh samples and record weight to the nearest ten-thousandth of a gram.
- 6.9 Record the volume of the test chamber in liters.
- 6.10 Plug in electronics and furnace units using AC outlet at alternate points of wall inlets.
- 6.11 Turn on power to weight transducer. Set recorders to standby. Allow 1 hour warmup.
- 6.12 Check temperature of water in water bottle around spring to ensure temperature is 45 degrees C \pm 1 degree C.
- 6.13 Turn on sparker power supply (not the sparker itself).
- 6.14 Calibration of weight transducer.
 - 6.14.1 Place an empty crucible in the stirrup.
 - 6.14.2 Zero the pen at any desirable point on the X-axis. Normally this is near the bottom of the graph.
 - 6.14.3 Set the scale and multiplier switches to a scale that will accommodate maximum sample weight loss. (Normally 250mg). Record both scale and multiplier selection used.
 - 6.14.4 Place a calibrating weight on the calibration weight pan that approximates the sample.
 - 6.14.5 Set the weight XY plotter to "on".
 - 6.14.6 Release the "calibration" knob lock and, if necessary, increase or decrease the setting to allow the desired change of scale.
 - 6.14.7 Remove the weights and rezero the Y-axis pen.
 - 6.14.8 Repeat 6 and 7 until desired weight change scale length is achieved.
NOTE: If the pointer stays off scale, move the transducer along the vertical axis of the column to bring the pointer in range.

- 6.14.9 Remove weights.
- 6.14.10 Lock calibration knob.
- 6.15 If a change of $\pm 10\text{mg}$ in the magnitude of either the weight or weight loss is recorded, the weight transducer should be calibrated. No changes to the zero point of the weight transducer are allowed after calibration.
- 6.16 Mount specimen in holder and place in TGA. Record type of holder.
- 6.17 Measure spark gap and distance between electrodes and material per 4.2.3.
- 6.18 If electrodes contact material surface during test run, the distance at the start of the test between electrodes and materials may be increased. Record the distance between electrodes and sample at start of test.
- 6.19 Thermocouple Calibration
 - 6.19.1 Place ice in ice reference "Thermos" junction.
 - 6.19.2 Set carriage scale and multipliers on both XY recorders to a value which will yield the desired scale length. Record both scale and multiplier selections used.
 - 6.19.3 Mark temperature values on graph paper at 10 div marks.
 - 6.19.4 Short the two "carriage" terminals on the TGA.
 - 6.19.5 Zero both units.
 - 6.19.6 Remove the shorting wire.
- 6.20 Photocell calibration
 - 6.20.1 Turn on water to cooling tubes and wait 5 minutes for temperatures to reach steady state.
 - 6.20.2 Connect the photocells to the bridge circuit and connect the bridge to the YX recorder.
 - 6.20.3 Place all shields and other equipment in test configuration. Raise the furnace.
 - 6.20.4 Turn off all unnecessary lights.
 - 6.20.5 Plug a galvanometer (or equiv.) into the recorder plug.
 - 6.20.6 Turn the bridge on and zero the meter.
 - 6.20.7 Adjust the zero on the photocell XY plotter to the desired point and set the scale to an appropriate value. Record both scale and multiplier selection used.

- 6.20.8 Test the set up by flashing and shining a flashlight up the furnace tube.
- 6.20.9 Turn off the bridge.
- 6.21 This calibration holds only for the physical surroundings present at test time. If lights or shields are changed, recalibration is necessary. Note whether recalibration was necessary.

7.0 TEST PROCEDURE

- 7.1 Purge the TGA sample chamber with a 40cc/min \pm 2cc/min gas flow for at least 5 minutes prior to the test. Record the gas or gas mixture designated by Test Preparation Sheet (TPS) as the test atmosphere.
- 7.2 Pressurize the TGA chamber to the pressure called out in the TPS. Record the pressure.
- 7.3 Allow gas or gas mixture to flow at rate specified in TPS. Record gas flow rate.
- 7.4 Heat TGA chamber at fixed rate. Record the rate at which the temperature is increased.
- 7.5 Record the temperature at which the start of weight loss occurred, as measured on the weight recorder by a discernible drop from the baseline.
- 7.6 Discharge the spark mechanism every minute beginning at ambient temperatures. Repeat until flash point (para.3.1) is reached or until 500 degrees C has been reached. Record flash point temperatures.
- 7.7 Continue the discharge of spark mechanism once each minute until the fire point is determined or 500 degrees C is reached. Record fire point.
- 7.8 Record the peak TGA chamber pressure.
- 7.9 Record the temperature at the completion of the test.
- 7.10 Record weight at end of test (immediately before fire point).

8.0 POST TEST PROCEDURE

- 8.1 Test shutdown
 - 8.1.1 As soon as visible flame appears shut down the gas supply and sparking mechanism. Shut off the photocells.
 - 8.1.2 Shut off the furnace controls.

- 8.1.3 After the fire has gone out, turn both XY recorders to standby.
- 8.1.4 Roll back the blast shield.
- 8.1.5 Open the door on the bottom of the furnace and lower it over the blower; turn the blower on.
- 8.1.6 Allow 40 minutes for the furnace and furnace tube to cool down.
- 8.1.7 Remove the furnace tube and clean with acetone or other appropriate solvent.
- 8.1.8 Blow the furnace tube out with compressed air (solvent fumes are explosive).
- 8.1.9 Remove the sample for further analysis.
- 8.1.10 Clean sparker electrode.

8.2 Calculations

- 8.2.1 Calculate weight loss.
- 8.2.2 Calculate percentage weight loss.
- 8.2.3 Calculate average percentage weight loss.
- 8.2.4 Calculate average flash point temperature in degrees C.
- 8.2.5 Calculate average fire point temperature in degrees C.
- 8.2.6 Calculate average temperature at the start of weight loss in degrees C.

8.3 Describe appearance of sample at end of test.

8.4 Observations and comments.

8.5 Sample residue and raw data received in bonded storage.

(state location of bonded storage)

9.0 REPORTING

Unless otherwise specified the following test data and pertinent information concerning the materials shall be reported.

9.1 Name of material (generic)

- 9.2 Vendor designation and vendor
- 9.3 Percentage weight loss
- 9.4 Flash point temperature
- 9.5 Fire point temperature
- 9.6 Temperature of start of weight loss
- 9.7 Atmosphere
- 9.8 Rate of temperature increase
- 9.9 Name and number of test procedure
- 9.10 Date of test
- 9.11 Test number
- 9.12 Identity of the testing agency or component
- 9.13 Name of test coordinator

SUPPLEMENTARY TEST NO. B
DIFFERENTIAL SCANNING CALORIMETER
SPARK IGNITION TEST

1.0 PURPOSE

This test procedure, when finalized, will provide a method for determining quickly and on small samples, the fire and flash points of nonmetallic materials used in the Apollo spacecraft.

(The procedure is currently under development)

SUPPLEMENTARY TEST NO. C

ELECTROCHEMICAL INITIATION OF WIRE INSULATION FIRE

1.0 PURPOSE

This test is designed to screen wire insulations and system coolant fluid combinations which may cause electrochemical initiation of fire. Wire insulations and coolant fluids used in this test shall be those which may be used in the habitable areas of manned spacecraft.

2.0 TEST DISCIPLINE

- 2.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 2.2 Each test shall have Quality Assurance Coverage and each step appropriately verified and stamped as required by the procedure.
- 2.3 Approval of the test conduct shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

3.0 TEST EQUIPMENT

- 3.1 Test Chamber - Test chamber shall have sufficient volume to insure complete combustion of the wire bundle specimen and shall be suitably constructed and protected to insure safe operation. A window or viewing port for visual observations shall be provided. The test chamber shall contain inlets for vacuum, air, oxygen, coolant fluid and a source of power. A horizontal sample holder shall be included and positioned within the test chamber.
- 3.2 Pressure Gauge - The pressure gauge shall be capable of measuring pressure up to 30 psia (1550 Torr) with an accuracy of ± 0.1 psia. A comparable pressure transducer may be used.
- 3.3 Oxygen Supply - The oxygen shall be commercially available oxygen conforming to specifications MIL-0-27210, Type 1. Suitable equipment for transferring oxygen to the test chamber shall be provided. A calibrated method of sampling the chamber atmosphere to verify percent oxygen is required.

- 3.4 Sample Holder - The sample holder shall consist of 4 vertically mounted ceramic insulators. They shall be in adjacent pairs located 13" apart, between which the wire samples are placed. The sample holder will be capable of rotating through varying angles with the horizontal.
- 3.5 Ignition Source - An external electrical power supply shall be provided which is capable of providing a controlled steady current through one or more wires of the test sample. The power source must be capable of supplying one-half the rated current of up to six AWG No. 20 wires.
- 3.6 Constant Drip Rate Apparatus - The constant drip rate mechanism shall include a tank, surgical tubing, a clamp valve attached to the tubing to regulate flow, and a No. 26 gauge surgical needle to yield uniform droplets.

4.0 DEFINITIONS

- 4.1 Standard Cut - A "standard cut" shall consist of a cut made through the outer covering or coverings of a wire and extending into the wire conductor. The cut shall not extend any deeper into the wire conductor than 1/4 of the nominal conductor diameter. All extraneous material shall be removed from the cut. The width of the cut shall be .030" ± .005.

5.0 SAMPLE PREPARATION

- 5.1 Wire Insulation Samples - Insulated wire samples shall be free of cuts, abrasions, or other flaws as determined by close visual observation. At the center of each test sample a standard cut as defined in 4.0 shall be made.
- 5.2 Additionally the samples shall be prepared in one of the following manners:
 - 5.2.1 Wire Insulation - Coolant Fluid Drip Test - Bundles of three or more wires shall be used. All wires shall have their standard cuts placed adjacent to and as close as possible to each other.
 - 5.2.2 Soaked Wire Test - Soak each sample in the coolant fluid for a period of one hour. Place the sample in a vacuum environment for a period of 12 hours.

5.2.3 Soaked Wire - Alcohol Test - Prepare per 5.2.2. Then wipe each sample with a cloth wet with isopropyl alcohol.

5.3 In all cases where bundles of wires are used, the standard cuts shall be placed adjacent to and as close as possible to each other. (See Fig. 1)

6.0 PRETEST PROCEDURE

6.1 Verify that all test equipment is in current calibration.

6.2 Verify oxygen certification (MIL-0-27210, Type 1)

6.3 Verify material identification

 6.3.1 Manufacturers Certification

 6.3.2 NASA Certification

 6.3.3 Contractor Certification

 6.3.4 Definite identification not available

6.4 Visually inspect each sample. (There shall be no cuts, abrasions or other flaws).

6.5 Prepare 3 specimens per 5.0. Note applicable number subparagraph of 5.0 to which the samples was prepared.

Verify standard cut per 4.1.

Note also the number of wires in bundles.

6.6 Record wire gauge number.

6.7 Record thickness of wire insulation over conductor and over shielding.

6.8 Record volume of chamber in liters.

6.9 Mount the test sample in the sample holder and place in test chamber.

6.10 Connect conductors and shields in either of the following configurations. Circle the configurations used in this test.

 6.10.1 All primary conductors shall be connected to the positive terminal of a 28 volt DC power supply and all shields to the negative terminal of the power supply. No current is to be flowing.

6.10.2 A 28 VDC power supply shall flow approximately 1/2 the rated current of the wire (3 amps for 20 AWG) through the primary conductor. All shields shall be connected to ground.

6.11 Arrangement in chamber. Circle arrangement used.

6.11.1 If samples are prepared per 5.2.1, the sample will be placed at an angle to the horizontal of between 5 degrees to 10 degrees. Record the angle _____ degrees. The damaged area will be kept wet by using the constant drip rate mechanism of 3.6. The droplets will be deposited above the cut area. Record the drop rate of this test.

6.11.2 If samples are prepared per 5.2.2 or 5.2.3 the sample shall be mounted horizontally.

7.0 TEST PROCEDURE

- 7.1 Evacuate the chamber to less than five (5) Torr and continue pumping the chamber for five (5) minutes.
- 7.2 Repressurize the chamber with oxygen to 16.5 ± 0.1 psia (854 Torr for the CM or 6.2 ± 0.1 psia (320 Torr) for the LM. Allow the chamber to stand for one minute. A leak is indicated if a decrease in test chamber of greater than 0.1 psia is observed after the system is closed off. If leak occurs, the chamber will be brought to atmospheric pressure, leak corrected, and steps 7.1 and 7.2 repeated
- 7.3 Analyze the chamber environment to verify that a minimum of 95.0% oxygen exists. Repurge or bake out chamber until this condition is met. (Record last oxygen percent reading.)
- 7.4 If 6.11.1 is chosen conduct test per 6.10.1 or 6.10.2 (current or no current). If 6.11.2 is chosen, after the chamber has stabilized at $16.5 \pm .1$ psia, flow tap water into the chamber pan until the bottom of the pan is covered to a depth of 1/4". Allow the chamber to set for 15 minutes.
- 7.5 Verify chamber pressure is at test pressure and isolate the chamber.
- 7.6 Conduct test per 6.10.1 or 6.10.2 (current or no current).
- 7.7 Record elapsed time from power supply "on" until ignition occurs.

8.0 POST TEST PROCEDURE

- 8.1 Observations (Bubbling, discolorations, sludge formations, smoke in sequential order denoting time of occurrence, etc.)
- 8.2 Comments
- 8.3 Sample residue and raw data received in bonded storage.

(state location of bonded storage)

9.0 REPORTING

Unless otherwise specified the following test data and pertinent information concerning the materials shall be reported.

- 9.1 Name of material
- 9.2 Specifications
- 9.3 Results of test including combustion phenomena.
- 9.4 Name and number of test procedure
- 9.5 Date of test
- 9.6 Test number
- 9.7 Identify of the testing agency or component
- 9.8 Name of test coordinator

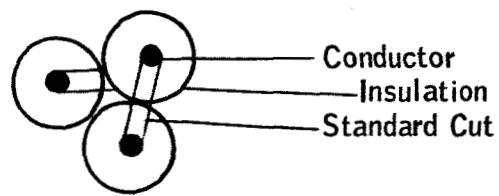


Figure 1
Typical Placement of Standard Cut in a
Three Wire Bundle

SUPPLEMENTARY TEST NO. D
AUTOGENOUS IGNITION POINT DETERMINATION

1.0 PURPOSE

This procedure is designed to screen materials used in habitable areas of the spacecraft which could start or promote the rapid spread of an accidental fire, at temperatures below 1000 degrees F.

2.0 TEST DISCIPLINE

- 2.1 Each test shall be directed by the cognizant Test Engineer or his appointed alternate.
- 2.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 2.3 Approval of the test conduct shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and the QA Inspector.

3.0 DEFINITION

- 3.1 **Fire Point** - The autogenous ignition point is the lowest temperature which the mixture of vapors from the surface of a materials and oxygen continue to burn after self ignition. The autogenous fire point will be determined by noting the temperature from thermo-couple readings.

4.0 TEST EQUIPMENT

4.1 Test Chamber -

- 4.1.1 **Construction:** The test chamber shall be suitably constructed to insure safe operation. A window or viewing port for visual observations shall be included. The chamber is to be fully protected against the possibility of operator injury in the event of explosive rupture.
- 4.1.2 No organic materials used in the construction shall be exposed to the interior atmosphere of the test chamber.

- 4.1.3 The test chamber shall have a volume sufficient to provide 12 liters per gram of sample materials.
- 4.1.4 Thermocouples - All temperature measurements made in the interior atmosphere of the test chamber shall be made using suitable thermocouples.
- 4.1.5 Specimen heater - A heating element capable of heating samples to at least 1000 degrees F \pm 25 degrees F is suitable for the purpose of this test. Modification of the furnace connections and wiring for safe operation in pure oxygen environments is required.
- 4.1.6 High Speed Motion Picture Camera - If a sample ignites, the remaining samples will be recorded on high speed film.
- 4.1.7 A recorder shall be provided to sense and record thermocouple readouts.
- 4.1.8 Pressure Gauge - A pressure gauge capable of measuring pressures from 0.1 to 30 psia with an accuracy of \pm 0.1 psia, or a pressure transducer and recorder with comparable capability shall be used.
- 4.1.9 Oxygen Supply - The oxygen shall be commercially available oxygen conforming to specification MIL-0-27210, Type 1. Suitable equipment for transferring oxygen to the test chamber shall be provided.

5.0 TEST SAMPLE PREPARATION

- 5.1 Test specimens will be tested as received, and preconditioned when specified on the TPS.
- 5.2 Test specimen will be 2" x 2" x thickness nearest to spacecraft usage. Foams will be 0.5" thick unless definite spacecraft thicknesses are known.
- 5.3 All tests will be run in triplicate.

6.0 PRETEST PROCEDURE

- 6.1 Verify that all test equipment is in current calibration.
- 6.2 Witness high speed movie camera setup.(If applicable per 7.6)
- 6.3 Witness thermocouple setup and checkout.
- 6.4 Verify oxygen certification (MIL-0-27210, Type 1)

- 6.5 Verify material identification
 - 6.5.1 Manufacturers Certification by one of the following.
 - 6.5.2 NASA Certification
 - 6.5.3 Contractor Certification
 - 6.5.4 Definite identification not available
- 6.6 Prepare three samples per paragraph 5.2 above.
- 6.7 If irregularly shaped samples are tested, describe the shapes.
- 6.8 Visually inspect each sample. (There shall be no cuts, abrasions or other flaws).
- 6.9 Clean samples by brushing or by flowing an inert gas to remove loose surface contamination.
- 6.10 Weigh the samples and record the weight.
- 6.11 Attach thermocouple to hot plate.
- 6.12 Place specimen on hot plate in the oxygen chamber.

7.0 TEST PROCEDURE

- 7.1 Seal chamber and evacuate to 0.25 psia or less and continue pumping for five minutes.
- 7.2 Isolate the chamber and monitor pressure for one (1) minute. Testing may not begin until all leaks are corrected. (A leak is indicated if an increase in pressure occurs)
- 7.3 Pressurize the chamber to 16.5, or 6.2 ± 0.1 psia for LM, with oxygen.
- 7.4 After the chamber has stabilized at the test pressure, soak the samples as follows: Ten (10) minutes for dense materials and thirty (30) minutes for porous materials. Record soak time.
- 7.5 Turn on the furnace and allow it to heat at the rate of 25°F per minute until ignition or a maximum of 1000°F is attained at which time the heat will be turned off.

- 7.6 Make visual observations for ignition. If ignition occurs, the remaining specimens will be run with camera coverage.
Note ignition or lack of ignition. Note temperature at which first observation of smoke occurred.
- 7.7 Observations (Include description of gases, smoke, etc.)
- 7.8 Burning phenomena (Color of flames, speed of burning, explosion, etc.)
- 7.9 Appearance of sample at test completion.
- 7.10 Record peak chamber pressure.
- 7.11 Turn off furnace and bleed off pressure to ambient condition.
Open chamber and remove specimens.

8.0 POST TEST PROCEDURES

- 8.1 Sample or residue and raw data received in bonded storage.

(state location of bonded storage)

- 8.2 Comments:

9.0 REPORTING

Unless otherwise specified the following test data and pertinent information concerning the materials shall be reported.

- 9.1 Name of material
- 9.2 Vendor Designation & Vendor
- 9.3 Results of Tests
- 9.4 Name & Number of test procedure
- 9.5 Date of Test
- 9.6 Identity of the testing agency
- 9.7 Name of test coordinator

SUPPLEMENTARY TEST NO. E

FRICITION AND IMPACT IGNITION TEST GUIDELINES

1.0 PURPOSE

This test procedure provides a method for determining the sensitivity and compatibility of nonmetallic materials with pure oxygen. The test is applicable to materials selected for use in the high pressure oxygen system. (Category D)

2.0 TEST CONDITIONS

- 2.1 The materials selected for the application shall have met the requirements of the acceptance tests designated in Category D of ASPO-RQ&TD-67-5A.
- 2.2 The materials shall be subjected to 20 successive tests at 70 foot-pounds (10 kilogram-meters) using the test procedure described in Section 7.0 below.
- 2.3 The pressure range of the GOX impact sensitivity testing shall be defined over the range of 20 psia to 1200 psia. For the purpose of this specification, a nonmetallic material specimen shall undergo GOX impact sensitivity testing at 20, 100, 500, 900 and 1200 psia. A new sample specimen shall be selected for each increased GOX pressure rating.

3.0 TEST DISCIPLINE

- 3.1 Each test shall be directed by the cognizant test engineer or his appointed alternate.
- 3.2 Each test shall have Quality Assurance coverage and each step appropriately verified and stamped as required by the procedure.
- 3.3 Approval of the test shall be indicated at the end of the test procedure by the signatures of the responsible Test Engineer and QA Inspector. Entries transcribed to the test data sheets will also be verified by the Test Engineer and QA Inspector.

4.0 CRITERIA OF ACCEPTABILITY

- 4.1 When tested according to the procedure in Section 7.0 of this document, the material shall show no reaction as indicated by:

- 4.1.1 Discoloration
- 4.1.2 Evidence of Combustion
- 4.1.3 Detonation
- 4.2 Materials showing a reaction at 500 psia shall be rejected and further testing discontinued.
- 4.3 Materials showing no reaction during the 20 tests at 900 and 1200 psia shall be accepted.
- 4.4 If there is more than one reaction at 900 and 1200 psia, the material shall be rejected.
- 4.5 If there is one reaction during the 20 tests, the materials shall be subjected to an additional 40 tests. If there is any reaction during the 40 tests, the material shall be rejected.

5.0 SAMPLE PREPARATION

- 5.1 Sufficient material shall be available to permit the preparation and testing of at least 60 specimen samples prepared as follows:
 - 5.1.1 Sheet materials up to 0.050 inch in thickness shall be tested as 0.250 inch diameter discs.
 - 5.1.2 Materials normally used in thickness greater than 0.250 inch shall be cut and tested as 0.250 inch diameter discs of $0.050 \pm .005$ thickness.
 - 5.1.3 Materials not readily available in sheet form, such as O-rings and the like, shall be tested in the available configuration, but the quantity used shall be limited to 0.5 ± 0.05 gram for each individual test.
 - 5.1.4 Materials such as greases and fluids whose thicknesses are not dictated by conditions of use shall be tested as $0.050 \pm .005$ inch layers in test cups.
 - 5.1.5 Coating materials such as dri-film lubricants shall be applied to 11/16 diameter stainless steel inserts in the same manner and to the same thickness that is intended for hardware application. After the samples have dried or cured, place in the regular specimen cups for test.

5.2 Sampling Cleaning

- 5.2.1 Soild materials, i.e., sheets, moldings, etc. shall be washed with a mild detergent solution, rinsed and dried by placing in a covered container in an air circulating oven at 212 to 239°F for one hour.
- 5.2.2 The test specimens shall be soaked in pure oxygen at room temperature for 24 hours in a separate pressure vessel.

6.0 PRETEST PROCEDURE

- 6.1 Verify that all the test equipment has been thoroughly cleaned according to the applicable method of the cleaning specification denoted in the procedure.
- 6.2 Verify that all test equipment is in current calibration.
- 6.3 Verify the material identification as one of the items below:
 - 6.3.1 Manufacturers Certification
 - 6.3.2 NASA Certification
 - 6.3.3 Contractor Certification
 - 6.3.4 Definite identification not available.
- 6.4 Observe that the cleaned metallic components and GOX transfer lines are assembled.
- 6.5 Verify that the sample is contained in a specimen holder and cup assembly which will allow for the transfer of impact energy to the subject specimen in a repitious manner.

7.0 TEST PROCEDURE

- 7.1 Place the sample in position within the sample retainer cavity.
- 7.2 Oxygen Generation - Moderate Pressure (20 psia to 2000 psia)
 - 7.1.1 Pass gaseous oxygen from manifold K-bottles through molecular sieve filters to separate oil, a check valve and an additional residual water filter (Drierite type) before final pressurization of the test chamber.

- 7.1.2 Set the plummet, pestle, striker or suitable impact device in position.
- 7.1.3 Raise the chamber pressure to the desired level by passing the oxygen through a suitable, stainless steel particle filter and thence into the test bomb.
- 7.3 Maintain the pressures in the test chamber at $\pm 10\%$ of the pressure selected.
- 7.4 Maintain the temperature at $70 \pm 2.0^{\circ}\text{F}$.
- 7.5 Activate the striker device.
- 7.6 Record the temperature and pressures in the chamber before, during and after the test.
- 7.7 At the conclusion of the test, release the residual GOX pressure in the assembly.
- 7.8 Disassemble the Apparatus.
- 7.9 Examine the sample specimen and cup specimen holder for visible signs of impact.
- 7.10 Record all observations.

8.0 REPORTING

The following test data and information shall be reported.

- 8.1 Name of material
- 8.2 Vendor and vendor designation
- 8.3 Results of test
 - 8.3.1 Pressure change in the vessel
 - 8.3.2 Temperature change in the vessel
 - 8.3.3 Condition of sample after test
 - 8.3.4 Condition of vessel after test

- 8.4 Name and number of test procedure
- 8.5 Date of test
- 8.6 Identity of testing agency
- 8.7 Name of test coordinator

SUPPLEMENTARY TEST NO. F
GAP PROPAGATION TEST

1.0 PURPOSE

This procedure describes the methods for determining the effectiveness of the gaps separating nonmetallic materials in the spacecraft in preventing a fire from propagating from one area to another.

(This procedure is currently under development.)

SUPPLEMENTARY TEST NO. G.
COMBUSTION TESTS CHARACTERISTICS

1.0 PURPOSE

This test is designed to measure the heat which a material will evolve during burning in a constant pressure of oxygen.

2.0 METHODS OF TESTING

Two methods of testing may be performed for heat of combustion:

- 2.1 Standard Method of Test for Heat of Combustion of Hydrocarbons by Bomb Calorimeter: 0-240-64.**
- 2.2 Testing by use of a Perkin-Elmer Differential Scanning Calorimeter - Model DSC-1B or equivalent.**

STANDARD METHOD OF TEST FOR
HEAT OF COMBUSTION OF HYDROCARBONS
BY BOMB CALORIMETER

1.0 TEST METHOD 2.1

2.0 SUMMARY OF METHOD

Heat of combustion is determined in this method by burning a weighed sample in an oxygen bomb calorimeter under controlled conditions. The heat of combustion is computed from temperature observations before, during, and after combustion, with proper allowance for thermochemical and heat transfer corrections. Either isothermal or adiabatic calorimeter jackets may be used.

3.0 DEFINITIONS AND UNITS

3.1 The energy units as used in this method are defined as follows:

3.1.1 1 cal (International Steam Table calorie) equals 4.1868 absolute joules.

3.1.2 1 BTU (British Thermal Unit) equals 251.996 cal equals 1055.07 absolute joules.

3.2 **Gross Heat of Combustion**, expressed as calories per gram or British Thermal Units per pound. - The heat released by the combustion of a unit mass of fuel in a constant volume bomb with substantially all of the water condensed to the liquid state.

3.3 **Net Heat of Combustion**, expressed as calories per gram or British Thermal Units per pound. - The heat released by the combustion of a unit mass of fuel at a constant pressure of 1 atmosphere with the water remaining in the vapor state (obtained by calculation from the determined gross heat of combustion.)

3.4 **Energy Equivalent** (effective heat capacity or water equivalent) of the calorimeter is the energy required to raise the temperature 1 deg, expressed as calories per deg. Cent. or deg. Fahr.

3.5 Temperatures may be measured in either Fahrenheit or Centigrade degrees (Note 1).

NOTE 1 - Temperatures may be recorded in ohms or other units when using electric thermometers. The same units must then be used in all calculations, including standardization.

3.6 Time is expressed in calculations in minutes and decimal fractions thereof. It may be measured in minutes and seconds.

3.7 Weights are measured in grams.

4.0 APPARATUS

Test Room, Bomb, Calorimeter, Jacket, Thermometers, and Accessories, as described in Appendix I.

5.0 REAGENTS

5.1 Alkali, Standard Solution (0.0725N). - Dissolve 3.84 g of Na_2CO_3 in water and dilute to 1 liter; alternatively, sodium hydroxide (NaOH) or potassium hydroxide (KOH) solutions of the prescribed normality are acceptable.

5.2 Benzoic Acid, Standard - Benzoic acid powder must be compressed into a tablet or pellet before weighing. Benzoic acid pellets for which the heat of combustion has been determined by comparison with the National Bureau of Standards sample are obtainable commercially for those laboratories not equipped to pellet benzoic acid.

5.3 Methyl Orange or Methyl Red Indicator.

5.4 Oxygen - Commercial oxygen produced from liquid air can generally be used without purification. If purification is necessary see Appendix I, Section A11.

5.5 2,2,4 - Trimethylpentane (Isooctane), Standard.

6.0 STANDARDIZATION

6.1 Determine the energy equivalent of the calorimeter as the average of not less than six tests using standard benzoic acid. These tests should be spaced over a period of not less than three days. Use not less than 0.9 g nor more than 1.1 g of standard benzoic acid ($\text{C}_6\text{H}_5\text{COOH}$). Make each determination according to the procedure described in Section 7 and compute the corrected temperature rise, t , as described in Section 8(8.1) or (8.2). Determine the

corrections for nitric acid (HNO_3) and firing wire as described in Section 8(8.3) and substitute in the following equation:

$$W = \frac{Hg + e_1 + e_3}{t}$$

where:

W = energy equivalent of calorimeter, in calories per degrees Fahrenheit or Centigrade,

H = heat of combustion of standard benzoic acid, 6318 cal per g,

g = weight of standard benzoic acid sample, in grams,

t = corrected temperature rise, as calculated in Section 8(a) or (b)

e_1 = correction for heat of formation of nitric acid, in calories, and

e_3 = correction for heat of combustion of firing wire, in calories.

Standardization tests should be repeated after changing any part of the calorimeter and occasionally as a check on both calorimeter and operating technique.

6.2 Checking the Calorimeter for Use with Volatile Fuels - Use National Bureau of Standards standard sample No. 217a (Section 5(e) to determine whether the results obtained agree with the certified value (20,541 Btu per lb, weight in air) within the repeatability of the method. If results do not come within this range, the method of handling the sample may have to be changed (Appendix I, Section A8). If this is not possible or does not correct the error, run a series of tests using standard sample No. 217a to establish the energy equivalent for use with volatile fuels.

7.0 PROCEDURE

7.1 Weight of Sample - Control the weight of sample (including any auxiliary fuel) so that the temperature rise produced by its combustion will be equal to that of 0.9 to 1.1 g of benzoic acid (Note 2). Weigh the sample to the nearest 0.1 mg.

NOTE 2 - If the approximate heat of combustion of the sample is known, the required weight can be estimated as follows:

$$g = \frac{11,373}{h_1}$$

where:

g = weight of sample, in grams, and

h_1 = approximate heat of combustion of sample, Btu per pound.

7.2 Water in Bomb. - Add 1.0 ml of water to the bomb from a pipet.

7.3 Oxygen. - With the test sample and fuse in place, charge the bomb with oxygen to 30 atmospheres gage pressure at room temperature (Note 3). Do not purge the bomb to remove entrapped air. CAUTION - Be careful not to overcharge the bomb. If, by accident, the oxygen introduced into the bomb should exceed 40 atmos, do not proceed with the combustion. An explosion might occur with possible violent rupture of the bomb. Detach the filling connection and exhaust the bomb in the usual manner. Discard the sample, unless it has lost no weight, as shown by reweighing.

NOTE 3. - Lower or higher initial oxygen pressures may be used within the range from 25 to 35 atmos, provided the same pressure is used for all tests, including standardization.

7.4 Calorimeter Water. - Adjust the calorimeter water temperature before weighing as follows:

Isothermal jacket
method (Paragraph

(e)) 3.0 to 3.5 F (1.6 to 2.0 C)
below jacket temperature
(Note 4).

Adiabatic jacket
method (paragraph

(f)) 2.0 to 2.5 F (1.0 to 1.4 C)
below room temperature.

Use the same amount (± 0.5 g) of distilled or deionized water in the calorimeter vessel for each test. The amount of water (2000 g is usual) can be most satisfactorily determined by weighing the calorimeter vessel and water together on a balance. The water may be measured volumetrically if it is measured always at the same temperature.

NOTE 4. - This initial adjustment will insure a final temperature slightly above that of the jacket for calorimeters having an energy equivalent of approximately 2450 cal per deg Cent. Some operators prefer a lower initial temperature so that the final temperature is slightly below that of the jacket. This procedure is acceptable, provided it is used in all tests, including standardization.

7.5 Observations - Isothermal Jacket Method. - Assemble the calorimeter in the jacket and start the stirrer. Allow 5 minutes for attainment of equilibrium; then record the calorimeter temperatures (Note 5) at 1-min intervals for 5 minutes. Fire the charge at the start of the sixth minute and record the time and temperature, t_a . Add to this temperature 60 percent of the expected temperature rise, and record the time at which the 60 percent point is reached (Note 6). After the rapid rise period (about 4 to 5 min), record temperatures at 1-min intervals on the minute until the difference between successive readings has been constant for 5 minutes.

NOTE 5. - Use a magnifier and estimate all readings (except those during the rapid rise period) to the nearest 0.005 F or 0.002 C when using ASTM Bomb Calorimeter Thermometers 56F or 56C. Estimate Beckman thermometer readings to the nearest 0.001 C and 25-ohm resistance thermometer readings to the nearest 0.0001 ohm. Tap mercurial thermometers with a pencil just before reading to avoid errors caused by mercury sticking to the walls of the capillary.

NOTE 6. - When the approximate expected rise is unknown, the time at which the temperature reaches 60 per cent of the total can be determined by recording temperatures at 45, 60, 75, 90, and 105 sec after firing and interpolating.

7.6 Observations - Adiabatic Jacket Method (Note 7). - Assemble the calorimeter in the jacket and start the stirrers. Adjust the jacket temperature to be equal to or slightly lower than the calorimeter, and run for 5 min to obtain equilibrium. Adjust the jacket temperature to match the calorimeter within ± 0.02 F (0.01 C) and hold for 3 min. Record the initial temperature (Note 5) and fire the charge. Adjust the jacket temperature to match that of the calorimeter during the period of rise, keeping the two temperatures as nearly equal as possible during the rapid rise, and adjusting to within ± 0.02 F (0.01 C) when approaching the final equilibrium temperature. Take calorimeter readings at 1-min intervals until the same temperature is observed in three successive readings. Record this as the final temperature. Time intervals are not recorded as they are not critical in the adiabatic method.

NOTE 7. - These instructions supersede the instructions given in Paragraph (e) when using jackets equipped for adiabatic temperature control.

7.7 Analysis of Bomb Contents. - Remove the bomb and release the pressure at a uniform rate such that the operation will require not less than 1 min. Examine the bomb interior for evidence of incomplete combustion. Discard the test if unburned sample or sooty deposits are found. Wash the interior of the bomb, including the electrodes and sample holder, with a fine jet of water and quantitatively collect the washings in a beaker. Use a minimum of wash water, preferably less than 350 ml. Titrate the washings with standard alkali solution, using methyl orange or methyl red indicator.

Remove and measure the combined pieces of unburned firing wire, and subtract from the original length. Record the difference as "wire consumed".

Determine the sulfur content of the sample if it exceeds 0.1 per cent. Determine sulfur by analyzing the bomb washings remaining after the acid titration, using the procedure described in ASTM Method D 129, Test for Sulfur in Petroleum Products by the Bomb Method.

8.0 CALCULATIONS

8.1 Temperature Rise in Isothermal Jacket Calorimeter. - Using data obtained as prescribed in Section 7(e), compute the temperature rise, t , in an isothermal jacket calorimeter as follows:

$$t = t_c - t_a - r_1(b - a) - r_2(c - b) \dots (1)$$

where:

t = corrected temperature rise,

a = time of firing,

b = time (to nearest 0.1 min) when the temperature rise reaches 60 per cent of total,

c = time at beginning of period in which the rate of temperature change with time has become constant (after combustion),

t_a = temperature at time of firing, corrected for thermometer error (Note 8),

t_c = temperature at time, c , corrected for thermometer error (Note 8),

r_1 = rate (temperature units per minute) at which temperature was rising during 5-min period before firing, and

r_2 = rate (temperature units per minute) at which temperature was rising during the 5-min period after time c . If the temperature is falling, r_2 is negative and the quantity $-r_2(c - b)$ is positive.

NOTE 8. - All mercury-in-glass thermometers must be corrected for scale error, using data from the thermometer certification prescribed in Appendix I, Section A5(a) or (b). Beckman thermometers also require a setting correction and an emergent stem correction (Appendix II, Section A12(b)). Solid-stem ASTM Thermometers 56F and 56 C do not require emergent stem corrections if all tests, including standardization, are performed within the same 10 F (5.5 C) interval. If operating temperatures exceed this limit, a differential emergent stem correction (Appendix II, Section A12 (a)) must be applied to the corrected temperature rise, t , in all tests, including standardization.

8.2 Temperature Rise in Adiabatic Jacket Calorimeter. - Using data obtained as prescribed in Section 7(f), compute the temperature rise, t , in an adiabatic jacket calorimeter as follows:

where:

t = corrected temperature rise,

t_a = temperature when charge was fired, corrected for thermometer error (Note 8), and

t_f = final equilibrium temperature, corrected for thermometer error (Note 8).

8.3 Thermochemical Corrections (Appendix II, Section A13). - Compute the following for each test:

e_1 = correction for heat of formation of nitric acid (HNO_3) in calories,

= milliliters standard alkali solution used in acid titration.

e_2 = correction for heat of formation of sulfuric acid (H_2SO_4), in calories,

$c_2 = 14 \times \text{percentage of sulfur in sample} \times \text{weight of sample in grams,}$

e_2 = correction for heat of combustion of firing wire in calories,

≈ 2.7 (centimeters of iron wire consumed)

$\equiv 2.7$ (centimeters of Nichrome wire consumed),
 $\equiv 2.3$ (centimeters of Chromel C wire consumed).

8.4 Gross Heat of Combustion. - Compute the gross heat of combustion by substituting in the following equation:

$$H_g = \frac{tW - e_1 - e_2 - e_3}{q} \quad \dots \dots (3)$$

where:

H_q = gross heat of combustion, in calories per gram,

t_c = corrected temperature rise as calculated in Section 8 (a) or (b),

W = energy equivalent of calorimeter, in calories per degrees

Fahrenheit or Centigrade (Section 6).

e_1, e_2, e_3 = corrections as prescribed in Paragraph (c), and

a = weight of sample in grams

9.0 REPORTING RESULTS

9.1 Usually the gross heat of combustion, H_g , in BTU per pound (obtained by multiplying H_g by 1.8) should be reported for fuel oils in preference to net heat of combustion, because of the difficulty in accurately determining the hydrogen content of the samples.

9.2 If the percentage of hydrogen, H , in the sample is known, the net heat of combustion may be calculated as follows:

where:

H_n = net heat of combustion, in Btu per pound,

H_q = gross heat of combustion, in calories per gram, and

H = hydrogen in the sample, in per cent.

9.3 If the percentage of hydrogen in aviation gasoline and turbine fuel samples is not known, the net heat of combustion may be calculated as follows:

$$H_n = 4310 + (0.7195)(1.8)(H_g) \dots \dots \dots (5)$$

where:

H_n = net heat of combustion, in Btu per pound, and

H_g = gross heat of combustion, in calories per gram.

1.0 PRECISION

The following data shall be used for judging the acceptability of results (95 percent probability):

10.1 **Repeatability.** - Duplicate results by the same operator should be considered suspect if they differ by more than the following amount:

Repeatability.....55BTU per lb.

10.3 Reproducibility. - Results submitted by two or more laboratories should be considered suspect if they differ by more than the following amount:

Reproducibility.....175 BTU per lb.

APPENDIX I

APPARATUS

A1. TEST ROOM

The room in which the calorimeter is operated must be free from drafts and not subject to sudden temperature changes. The direct rays of the sun shall not strike the jacket or thermometers. Adequate facilities for lighting, heating, and ventilating shall be provided. Thermostatic control of room temperature, and controlled relative humidity are desirable.

A2. OXYGEN BOMB

The oxygen bomb shall have an internal volume of 350 ± 50 ml. All parts shall be constructed of materials which are not affected by the combustion process or products sufficiently to introduce measurable heat input or alteration of end products. If the bomb is lined with platinum or gold, all openings shall be sealed to prevent combustion products from reaching the base metal. The bomb must be designed so that all liquid combustion products can be completely recovered by washing the inner surfaces. There must be no gas leakage during a test. The bomb must be capable of withstanding a hydrostatic pressure test to 3,000 psig at room temperature without stressing any part beyond its elastic limit.

A3. CALORIMETER

The calorimeter (Note 9) vessel shall be made of metal (preferable copper or brass) with a tarnish-resistant coating, and with all other surfaces highly polished. Its size shall be such that the bomb will be completely immersed in water when the calorimeter is assembled. It shall have a device for stirring the water thoroughly and at a uniform rate, but with minimum heat input. Continuous stirring for 10 min shall not raise the calorimeter temperature more than 0.01 C starting with identical temperatures in the calorimeter, room, and jacket. The immersed portion of the stirrer shall be coupled to the outside through a material of low heat conductivity.

NOTE 9

As used in this method, the term "Calorimeter" designates the bomb, the vessel with stirrer, and the water in which the bomb is immersed.

A4. JACKET

The calorimeter shall be completely enclosed within a stirred water jacket and supported so that its sides, top, and bottom are approximately 1 cm from the jacket walls. The jacket may be arranged so as to remain at substantially constant temperature, or with provision for rapidly adjusting the jacket temperature to equal that of the calorimeter for adiabatic operation. It must be constructed so that any water evaporating from the jacket will not condense on the calorimeter.

A double-walled jacket with a dead-air insulation space may be substituted for the constant-temperature water jacket if the calorimeter is operated in a constant-temperature (± 2 F) room. The same ambient conditions must be maintained for all experiments, including standardization.

A5. THERMOMETERS

Temperatures in the calorimeter and jacket shall be measured with the following thermometers or combinations thereof:

- (a) Etched Stem, Mercury-in-Glass, ASTM Bomb Calorimeter Thermometer having a range of 66 to 95 F or 19 to 35 C, as specified, and conforming to the requirements for thermometer 56 F or 56C, respectively, as prescribed in ASTM Specifications E1. Each of these thermometers shall have been tested for accuracy at intervals no larger than 2.5 F or 2.0 C over the entire graduated scale. Corrections shall be reported to 0.005 F or 0.002 C, respectively, for each test point.
- (b) Beckman Differential Thermometer, range approximately 6C in 0.01 subdivisions reading upward, tested for accuracy at intervals no larger than 1 C over the entire graduated scale and corrections reported to 0.001 C for each test point.
- (c) Calorimetric Type Platinum Resistance Thermometer, 25-ohm.

A6. THERMOMETER ACCESSORIES

A magnifier is required for reading mercury-in-glass thermometers to one-tenth of the smallest scale division. This shall have a lens and holder designed so as not to introduce significant errors due to parallax.

A wheatstone bridge and galvanometer capable of measuring resistance to 0.0001 ohm are necessary for use with resistance thermometers.

A7. TIMING DEVICE

A watch or other timing device capable of measuring time to 1 sec is required for use with the isothermal jacket calorimeter.

A8. SAMPLE HOLDER

Nonvolatile samples shall be burned in an open crucible of platinum (Preferred), quartz or acceptable base metal alloy. Base metal alloy crucibles are acceptable if after a few preliminary firings the weight does not change significantly between tests.

Volatile samples shall be sealed in thin-walled glass bulbs (preferred), in metal sample holders sealed with cellulose tape, or in gelatin capsules, following acceptable procedures for handling such liquids.

A9. FIRING WIRE

Use a 10-cm length of No. 34 B & S gage iron wire or Chromel "C" resistance wire. Shorter lengths may be used if the same length is employed in all tests, including standardization tests. Platinum wire may be used if the ignition energy is small and reproducible.

A10. FIRING CIRCUIT

A 6 to 16-v alternating or direct current is required for ignition purposes with an ammeter or pilot light in the circuit to indicate when current is flowing. A step-down transformer connected to a 115-v 50/60 cycle lighting circuit or storage batteries may be used.

Caution - The ignition circuit switch shall be of the momentary contact type, normally open, except when held closed by the operator.

A11. OXYGEN PURIFYING DEVICE

Commercial oxygen produced from liquid air can generally be used without purification. Oxygen prepared by electrolysis of water should not be used without purification, as it may contain enough hydrogen to affect results by 1 per cent or more. Combustible impurities may be removed from oxygen by passing it over copper oxide (CuO) at about 500 C.

APPENDIX II

A12. THERMOMETER CORRECTIONS

(a) The differential emergent stem correction for solid stem calorimetric thermometers (56 F and 56 C) may be computed from the following formula:

Differential stem correction

$$= K (t_c - t_a - (t_c + t_a - L - T))$$

where:

K = differential expansion coefficient of mercury in glass = 0.00016 for Centigrade thermometers or 0.00009 for Fahrenheit thermometers,

L = scale reading to which the thermometer was immersed,

T = mean temperature of emergent stem,

t_c = Initial temperature reading, and

t_a = final temperature reading.

TABLE I - CORRECTION FACTORS

"Setting"	Factor
15.....	-0.0015
20.....	0.0000
25.....	+0.0015
30.....	+0.0029
35.....	+0.0043
40.....	+0.0056

(b) Differential emergent stem correction for a Beckman thermometer immersed to the zero of the scale may be computed as follows:

Differential stem correction

$$= K (t_c - t_a (S + t_c + t_a - T))$$

where:

S = the "setting" (temperature at zero reading) of the thermometer, K, T, t_c and t_a as defined in Paragraph (a).

(c) "Setting" correction for a Beckman thermometer may be computed as follows:

$$\text{"Setting" correction} = \text{factor} X (t_c - t_a)$$

where:

t_c and t_a are as defined in Paragraph (a), and the factor is as obtained from Table I.

A13. THERMOCHEMICAL CORRECTIONS

- (a) Heat of Formation of Nitric Acid. — A correction of 1 cal is applied for each milliliter of standard alkali used in the acid titration. This is based upon the assumption that (1) all of the acid titrated is nitric acid (HNO_3), and (2) that the heat of formation of 0.1 N HNO_3 under bomb conditions is 13.8 kg-cal per mol. When sulfuric acid (H_2SO_4) is also present, part of the correction for H_2SO_4 is contained in the e_1 correction (Section 8 (c)) and the remainder in the e_2 correction (Section 8 (c)).
- (b) Heat of Formation of Sulfuric Acid. — A correction of 1.4 kg-cal is applied for each gram of sulfur in the sample. This is based upon the heat of formation of 0.17 N H_2SO_4 which is - 72 kg-cal per mol. But, a correction equal to 2×13.8 kg-cal per mol of sulfur was applied for H_2SO_4 in the e_1 calculation (Section 8 (c)). So the additional correction necessary is $72 - (2 \times 13.8) = 44.4$ kg-cal per mol or 1.4 kg-cal per gram of sulfur.

The value of 1.4 kg-cal per gram of sulfur is based on a fuel oil containing a relatively large amount of sulfur since as the percentage of sulfur decreases, the correction decreases and consequently a larger error can be tolerated. For this calculation 0.8 per cent, S, 99.2 per cent CH_2 was taken as the empirical composition of fuel oil. If a 0.6-g sample of such a fuel oil is burned in a bomb containing 1 ml of water, the sulfuric acid formed will be approximately 0.17 N H_2SO_4 . Using data from National Bureau of Standards Circular No. 500, the heat of reaction SO_2 (g) + $1 \frac{1}{2} O_2$ (g) + $651 H_2$ (l) = H_2SO_4 . $650 H_2O$ (l) at constant volume and 30 atmos is - 72 kg-cal per mol.

- (c) Heat of Combustion of Fuse Wire. — The following heats of combustion are accepted:

Iron wire, No. 34 B&S gage = 2.7 cal per cm Chromel C wire, No. 34

B&S Gage = 2.3 cal per cm.